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Final Report to United States Army Toxic and Hazardous Materials Agency December 1987



Development of a New Process for Treatment of Paint Sludge Wastes

(Task Order Number 6)

Final Report

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The amount of ash generated after thermal treatment was in the range of 36 to 55% of the original weight of the air dried paint samples. The elements of most concern with regard to TCLP criteria appear to be barium (in the epoxy primer) and chromium (in the green pigments). Thermal treatment of all the samples in the temperature range of 700 to 1000°C generated ashes which passed the TCLP procedure. A large effect of temperature on the concentration of barium and chromium in the extracts was noted. The ash generated at 1000°C from the epoxy primer gave barium levels in the TCLP extracts which were 25 to 43% of the regulatory criteria. The ash generated at 1000°C from the green CARC paints gave chromium levels in the TCLP extracts which were 23 to 31% of the regulatory criteria.

Thermal treatment of the epoxy primer ash to a temperature of 1100°C produced an ash which failed the TCLP criteria for barium. Similar treatment of the green CARC paint ash increased the extract chromium levels to 74% of the regulatory criteria. The epoxy primer ash when subjected to 1450°C produced zinc oxide crystals which volatilized from the ash. The results of glassification experiments with the epoxy primer ash indicated the following: • Sodium carbonate (11%) as the only additive was not effective; • Phosphate frit additives (50%) were not effective; and • Sodium carbonate (11%) and silica (33%) additives were effective in reducing the extractable barium.

The results of the glassification experiments with the green CARC ash indicated the following: • Sodium carbonate (11%) and silica (33%) additives were not effective in incorporating the chromium; and • Phosphate frit additives (50%) were slightly better than the sodicarbonate and silicate additives but still did not generate a non-hazardous residue.

The results of the glassification experiments under reducing conditions (with carbon as additive) indicated the following: • The addition of sodium carbonate (11%), silica (33%), and carbon (18%) to the epoxy primer ash generated a non-hazardous glass and a metal phase composed primarily of iron and some phosphorus; • The addition of similar additives to the green CARC ash generated a non-hazardous glass phase and a metal phase composed primarily of chromium and iron with some cobalt; and • The generated products (glass and metal phases) for both the epoxy primer and green CARC ashes yielded TCLP extracts which passed the regularory criteria.

The results of the laboratory program suggest that thermal treatment of paint waste under some conditions may be feasible for generation of non-hazardous ash residue. The results also indicate that thermal treatment (reducing conditions) is feasible for generation of a non-hazardous inorganic glass phase and a metal phase which contains the three metals of interest (chromium, cobalt and iron). In addition, zinc present in the waste may be recovere via volatilization and subsequent condensation, if so desired. Further experiments on a pilot-scale are recommended, however, to investigate these approaches to determine the need for subsequent treatment (e.g., glassification and/or recovery) of the ash product and the actual destruction efficiency of organic components.

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1.0 SUMMARY

This report presents the results of laboratory tests performed on paint waste samples obtained from the Letterkenny Army Depot (LEAD). The purpose of these tests was to determine if the ash residue from a thermal treatment process such as combustion would be classified as hazardous according to the Environmental Protection Agency (EPA) Toxicity Characteristic Leaching Procedure (TCLP). In addition, the feasibility of generating a glassified product from the ash which would be classified as non-hazardous was also tested. Finally, tests were also performed to determine if recovery of selected metals from the ash is feasible.

The choice of samples tested was based upon information received from LEAD as to the type, quantity and relative importance of the various paint wastes generated. The samples tested included a spent spray booth filter, epoxy primer, five shades of Chemical Agent Resistant Coatings (CARC) paint and three shades of epoxy/polyamide paint.

The candidate treatment processes tested included thermal treatment (combustion) of the paint waste samples in the 700 to 1000°C range and at heating rates of 0.2 to 0.5 and 3.3 to 7 deg/sec, followed by: (1) elevated temperature (1100 and 1450°C) treatment of selected ash residues; (2) glassification of the ash with phosphate and silicate additives; or (3) glassification of selected ash residues with silicate additives followed by simultaneous separation of metals from the ash.

The data collected included: amount of weight loss, both upon sample preparation (air drying) and after thermal treatment (combustion) of the paint waste samples; composition (29 elements) of the ash residue generated after combustion; and concentration of elements (29) in TCLP extracts of the various ashes and major components of the glassified products and composition of the separated metal phases.

The amount of ash generated after thermal treatment (combustion) was in the range of 36 to 55% of the original weight of the air dried paint samples. The ash produced by the epoxy primer was high in barium, zinc and iron and contained phosphorous. The CARC paints contained (depending on color) high amounts of chromium with moderate amounts of iron, cobalt and zinc. Only the green shade of the epoxy/polyamide paint contained smaller amounts of chromium and iron. Other major components of the ashes generated were typically silicon, magnesium and titanium (mostly in the epoxy/polyamide paint). The elements of most concern with regard to TCLP criteria appear to be barium (in the epoxy primer) and chromium (in the green CARC paints). The elements of most interest from a recovery standpoint appear to be zinc (in the epoxy primer) and chromium and cobalt (in the green pigments).

Thermal treatment (combustion) of all the samples in the temperature range of 700 to 1000°C generated ashes which passed the TCLP procedure. A large effect of temperature on the concentrations of barium and chromium in the extracts was noted. The ash generated at 1000°C

from the epoxy primer gave barium levels in the TCLP extracts which were 25 to 43% of the regulatory criteria. The ash generated at 1000°C from the green CARC paints gave chromium levels in the TCLP extracts which were 23 to 31% of the regulatory criteria. No other effects of temperature or heating rate were noted.

Thermal treatment of the epoxy primer ash to a temperature of 1100°C produced an ash which failed the TCLP criteria for barium. Similar treatment of the green CARC paint ash increased the extract chromium levels to 74% of the regulatory criteria. Decomposition of barium and chromium compounds at the elevated temperatures (possibly aided by the presence of oxygen) explain the observed results. These data suggest that thermal treatment (combustion) of the paint wastes at these elevated temperatures cannot guarantee the production of a non-hazardous product.

The epoxy primer ash when subjected to 1450°C produced zinc oxide crystals which volatilized from the ash.

The results of glassification experiments with the epoxy primer ash indicated the following:

- · Sodium carbonate (11%) as the only additive was not effective;
- Phosphats frit additives (50%) were not effective; and
- Sodium carbonate (11%) and silica (33%) additives were effective in reducing the extractable barium.

The results of the glassification experiments with the green CARC ash indicated the following:

- Sodium carbonate (11%) and silica (33%) additives were not effective in incorporating the chromium:
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The results of the glassification experiments under reducing conditions (with carbon as additive) indicated the following:

- The addition of sodium carbonate (11%), silica (33%) and carbon (18%) to the epoxy primer ash generated a non-hazardous glass and a metal phase composed primarily of iron and some phosphorus,
- The addition of similar additives to the green CARC ash generated a non-hazardous glass phase and a metal phase composed primarily of chromium and iron with some cobalt; and
- The generated products (glass and metal phases) for both the epoxy primer and green CARC ashes yielded TCL? extracts which passed the regulatory criteria.

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The results of the laboratory tests also indicate that thermal treatment (reducing conditions) is feasible for generation of a non-hazardous inorganic glass phase and a metal phase which contains the three metals of interest (chromium, cobalt and iron). In addition, zinc present in the waste may be recovered via volatilization and subsequent condensation, if so desired. Consequently, it is recommended that depending upon the results obtained from thermal treatment pilot-scale tests, further investigation of this approach (thermal treatment under reducing conditions followed by glassification and recovery of metals) also be tested on a pilot-scale to determine destruction efficiency for organics as well as detailed product characteristics.

2.0 BACKGROUND AND OBJECTIVES

Many U.S. Army depot operations involve both the removal and application of paints. These processes generate a variety of paint containing wastes that require treatment and/or disposal. These paint wastes contain organic and inorganic compounds that can classify them as a hazardous waste. Current disposal practice for these wastes generally consists of landfilling in a hazardous waste disposal site. This type of disposal for wastes which contain volatile and combustible materials is likely to be restricted in the future. Consequently, changing regulations provide an impetus for investigating methods and processes which can potentially reduce the volume of the hazardous waste and/or render it non-hazardous.

Presently, a variety of technologies are available for treating hazardous waste. However, the applicability of these technologies to paint containing waster is largely untested. Consequently, the overall objective of this program was to perform tests on selected samples of paint wastes so as to determine if a selected treatment process is likely to generate a non-hazardous material.

The approach utilized in this program to address the overall objective consisted of the following steps:

- Visiting a typical military installation (LEAD) involved with painting/depainting operations to obtain information on the quantity and type of materials (paints, solvents, blast media, etc.) used and wastes generated;
- Selection of paint samples to be tested based upon this data;
- Selection of a candidate treatment technology;
- Acquisition of paint samples from LEAD and associated suppliers;
- Performance of laboratory thermal treatment and additive tests; and
- Ferformance of chemical analyses and EPA TCLP tests on treated paint wastes samples.

3.0 PAINT UTILIZATION AND WASTE DISPOSAL AT LETTERKENNY ARMY DEPOT

3.1 Consumption of Paints and Solvents

As part of the process of selecting paint waste samples to be tested, a visit was made to LEAD (Chambersburg, PA) along with the request for information regarding the paint and solvent consumption at the depot. A summary of the volume of the various paints and solvents consumed at this depot for two typical months (April and May 1986) is given in Table 3-1.

The consumption of paints and solvents at LEAD can be classified into five categories. These are:

- (1) Primers (generally epoxies),
- (2) Chemical Agent Resistant Coatings (CARC) consisting of polyurethane paints.
- (3) Other paints (such as epoxy/polyamides),
- (4) Solvents (such as methyl ethyl ketone (MEK) and other thinners), and
- (5) Miscellaneous materials (such as water).

Of these five categories the CARC paints, primers and solvents represented the majority of the materials consumed.

3.2 Generation of Paint Wastes

The variety of solid wastes generated in the painting and depainting operations at LEAD can also be classified into categories. These are:

- (1) Spent blast media wastes,
- (2) Excess paint and solvent residues,
- (3) Spent spray booth filters, and
- (4) Spent solvent strippers/paint sludges.

The spent blast media wastes result from stripping various equipment items, to be refurbished, using sand, walnut shells, and steel shot. Future operations may include the use of plastic media for stripping and thus will generate a waste containing this material. The spent solvent stripper and paint sludge is generated from chemical stripping of paint from items that cannot be stripped by blasting. The excess paint and solvent residues result from both painting operations and the cleaning of painting equipment during and at the end of the various shifts. The spent spray booth filters result from the periodic replacement of these filters located in the paint spray booths.

A summary of the estimated paint waste generation at LEAD is given in Table 3-2. These data indicate the majority of the waste paint remains within the spent spray booth filters and spent blast media followed by smaller quantities in the paint and solvent mixed residues and spent solvent strippers/paint sludges.

TABLE 3-1

CONSUMPTION OF PAINTS AND SOLVENTS
AT LETTERKENNY ARMY DEPOT

	April 1986		May 1986		
Category	Consumption (gallons)	Percent of Total	Consumption (gallons)	Percent of Total	
Primers (epoxy) ^a	329	13	350	13	
CARC ^b	1102	44	1179	44	
Other Paints	327	13	231	9	
Solvents(Thinners)					
MEK	278	11	255	10	
Others	391	16	269	10	
Miscellaneous ^e	66	3	73	3	
Other (water)	0	0	283	11	
		-	-		
Total	2493	100	2640	100	

Source: Letterkenny Army Depot

^aPigments include red, white, green and zinc chromate in minor amounts.

Pigments include forest green, sand, black, green, brown, aircraft red and white.

CPigments include forest green, sand, gloss gray, white, yellow and red.
 (in epoxy/polyamides and enamels)

dMEK - Methyl ethyl ketone. Other solvents listed include isopropyl alcohol, aliphatic thinner, dope and lacquer thinner and denflex thinner.

^eMiscellaneous items include catalyst, lubricant, battery box coating, walkway compound, acid wash pretreatment.

TABLE 3-2 ESTIMATED PAINT WASTE GENERATION AT AT LETTERKENNEY ARMY DEPOT

Waste Type Qua	ntity Generated <u>Distribut</u>		bution
		Paint(gal/month)	Other(gal/month)
Spent Plast Media	1000 to 1300	170	4400(Blast media)
Waste Paint/Solvent	s 600	45	2700 (Solvents)
Spent Spray Booth Filters	360 to 480	300	NA (Filters)
Spent Chemical Strippers/Paint Sludge	100	NA	460(Mixture)

NA - Not Available

Source: Arthur D. Little, Inc., based on information provided by Letterkenny Army Depot.

It is important to note that the paint and primer coatings being removed from the equipment to be refurbished is different from that presently being applied at the depot. Current paint operations call for use of CARC paints and epoxy primers which are chromium and lead free whereas the paint being removed contains zinc chromate primers and non-polyurethane paint. The chemical composition of the paint waste (spent blast media and chemical stripper sludge) from depainting operations is thus very different from the chemical composition of the paint waste from painting operations (paint and solvent residues and spent spray booth filters).

3.3 Treatment and Disposal of Paint Waste

The current treatment and disposal practices at LEAD for the aforementioned types of wastes is as follows:

- Spent Blast Media Wastes: drummed and stored on-site.
- Paint and Solvent Residues:
 - "Clean" solvent supernate decanted for subsequent offsite incineration,
 - Sludge residue mixed with cement kiln dust, solidified and landfilled off-site,
 - Hardened solid paint residues directly landfilled off-site.
- Spent Spray Booth Filters: mixed with cement dust, solidified, and landfilled off-site.
- Spent Solvent Strippers and Paint Sludge: trucked off-site for subsequent incineration or landfilling.

All landfilling is off-site, at hazardous waste disposal facilities. It appears that the current disposal practices utilized are greatly weighted towards landfilling with some treatment and no recovery; practices which most likely will require some modification in order to keep in line with the Department of Defense (DOD) hazardous waste minimization goals and anticipated regulations further restricting landfilling as an acceptable method of disposal.

4.0 SELECTION OF PAINT SAMPLES AND CANDIDATE TREATMENT PROCESSES

4.1 Paint Samples

The selection of paint wastes to be studied under this test program was based on the following criteria:

- Relative ranking of importance of the waste categories by LEAD personnel;
- Current volume of waste generated and likelihood that that volume will change in the future;
- The relative hazardous nature of the waste;
- The relative impact that future regulations are likely to have on the current waste disposal practices; and
- · Funding limitation of the program.

Letterkenny personnel ranked the paint and solvent residues and the spent spray booth filters high on their list of importance. As the data in Table 3-2 indicate, the waste category with highest volume being disposed is that of the spent blast media, followed by the spent spray booth filters and paint solvent residue. Future consideration of volume however, indicated that the spent blast media will likely be reduced duo to use of plastic media and media recycling while the spent spray booth filters and paint solvent residues will remain constant. In addition, since painting is now done with CARC paint, new items in the future that will be stripped will contain the new paint formulation and a decreasing amount of the old paint formulation. Due to the high organic solvent content of the spray booth filters and paint/solvent residue these are classified as more toxic than the spent blast media which contain mostly blast medium material mixed with dried paint. Similarly, since the paint-solvent residues and spent spray booth filters contain volatile and flammable materials which are likely to be restricted from landfilling in future regulations, these rank high on the regulatory impact criteria.

A summary of the relative ranking of the waste paint categories is given in Table 4-1.

Based on these criteria the selected waste samples were in the following two categories:

· Spent spray booth filters, and

T

 Waste paints/solvents (CARC, epoxy/polyamide paint and epoxy primer).

The spent spray booth filter samples were selected since they would represent a mixture of the paints currently being applied in depot spraying operations.

TABLE 4-1

RELATIVE RANKING OF PRIORITY FOR VARIOUS
PAINT WASTE CATEGORIES

Ranking Criteria Sp		Paint Waste Category				
		Spent Blast Media	Waste Paint/ Solvents	Spent Spray Booth Filters	Spent Chemical Strippers/ Paint Sludge	
						
Depot Co	ncern	Low	High	High	Low	
Current Volume G	Waste eneration	High	Medium	Medium	Lew	
Future W Generati	aste Volume on	Medium	Medium	Medium	Low	
Quantity of Hazardous Components		s Low	High	High	High	
Anticipated Impact of Future Regulations on Current Disposal		Medium s	High	High	High	
Method					-	
TOTAL:	High Medium Low	1 2 2	3 2 0	3 2 0	2 0 3	
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Source: Arthur D. Little, Inc.

In the category of the waste paints/solvents three major components are the most currently used: CARC, epoxy primer and the epoxy/polyamide materials. Currently, the CARC paint being used is a one component, moisture (air) cured material. In constrast, the epoxy primer currently used is a two component system as are also the epoxy/polyamide paints.

The focus of this program was on the inorganic components, the colors (pigments) which are incorporated into the waste materials. The inorganic pigments that are currently being utilized in the various colors are given in Table 4-2. The chemical composition of these pigments are summarized in Table 4-3.

The spent spray booth filters may contain a mixture of all the materials being sprayed which include the solvents, primer, CARC and other paint materials used in low quantities such as epoxy/polyamides and enamels. This sample represents an actual disposal situation where all the pigments are mixed. The primer being utilized is primarily the red epoxy material (brown epoxy is sometimes also used). The CARC paints have the greatest diversity in pigments that may be able to be studied separately. Based upon this information, the following samples were selected for study:

- · Spent spray booth filter,
- · Epoxy primer,
- CARC 383 Green,
- . CARC Forest Green,
- CARC 383 Sand,
- CARC 383 Brown,
- CARC 383 Black,
- Epoxy/polyamide seafoam green
- Epoxy/polyamide gray, and
- Epoxy/polyamide white.

4.2 Candidate Treatment Processes

A matrix of possible treatment options for the paint wastes is shown in Table 4-4. These treatment options have been categorized into three levels. The first level represents processes which result in the separation of the organic and inorganic fractions except for thermal treatment or combustion which simultaneously also treats (destroys) the hazardous nature of the organic fraction. The second level involves processes which remove or fix the hazardous components in the separated fractions. The third level involves processes which concentrate the hazardous or desirable inorganic component from the phase produced in the second level. The options shown are simplified since they do not include processes which are capable of simultaneously performing several of these functions, such as a thermal process which destroys the organics, generates a glass product and recovers some metal(s) simultaneously.

Selection of a waste treatment process for the paint wastes needs to consider the following factors:

TABLE 4-2

EXPECTED COMPOSITIONS FOR VARIOUS INORGANIC PIGMENTS
IN THE INORGANIC FRACTION OF PAINT WASTE

Color	Pigment	Composition (%)
Epoxy Primer	Red Iron Oxide Zn Phosphate Silica Mg Silicate	20-45 10-25 20-45 0-25
Green Shades	Acid Insoluble Green Yellow Iron Oxide Red Iron Oxide Mg/Fe Oxide Silica Mg Silicate Organic	20-35 10-20 0-5 0-10 30-60 0-30
Sand/Yellow/ Drab	Yellow Iron Oxide Red Iron Oxide Chromium Oxide Titanium Dioxide Silica Mg Silicate Organic	8-25 1-5 4-30 1-25 25-67 0-30
Blacks/ Some Greens	Black Iron Oxide Red Iron Oxide Yellow Iron Oxide Silica Mg Silicate Organic	0-48 0-26 0-5 40-83 0-40
Browns/Reds	Yellow Iron Oxide Red Iron Oxide Titanium Dioxide Brown Iron Oxide Silica Mg Silicate Organic	5-37 0-7 0-5 0-10 25-50 0-25
Whites	Titanium dioxide Silica Mg Silicate	0-60 25-70 0-30
Grays	Titanium Dioxide Yellow Iron Oxide Organic	0-50 0-50

Carbazole Dioxazine Violet Carbon Black

Source: MIL-C-46168A(MR) and MIL-C-53039(ME)

TABLE 4-3

CHEMICAL COMPOSITION OF INORGANIC PIGMENTS AND EXTENDERS USED IN VARIOUS PAINT COLORS

Pigment/Extender	Chemical Formula
Acid Insoluble Green	Mixture of $Co_2O_3/2nO/Cr_2O_3$
Yellow Iron Oxide	Fe ₂ 0 ₃ .H ₂ 0
Iron Oxide (Red/Brown)	Fe ₂ 0 ₃
Magnesium/Iron Oxide	MgO.Fe ₂ O ₃
Silica	sio ₂
Magnesium Silicate	MgO.SiO ₂
Chromium Oxide	Cr ₂ 0 ₃
Black Iron Oxide	Fe ₃ 0 ₄
Titanium Dioxide	Tio ₂
Zinc Phosphate	Zu ₃ (PO ₄) ₂

Note: Organic pigments also usually associated with the various commonly used colors are carbon black (C) and Carbazole Dioxazine Violet. Additional filler materials may exist such as Barium Sulfate.

Source: Arthur D. Little, Inc.

TABLE 4-4

OPTIONS FOR TREATMENT OF PAINT WASTE

Level		Description	<u>M</u>	<u>lethods</u>
I	1.	Separation of Inorganic and Organic Phases	a. b. c. d.	Digestion Solubilization
II	1.	Removal or Fixation of Hazardous Species in Separated Inorganic Residue (From Level I)	a. b.	Vitrification (during combustion) Vitrification (post combustion) Solubilization (e.g. H ₂ SO ₄)
	2.	Treatment of Hazardous Species in Separated Organic Residue (From Lavel I)	a. b.	Combustion/chermal treatment Biological Digestion
III	3.	Concentration of Extracted Hazardous Inorganics From Separated Inorganic Residue (From Level II)	a. b. c. d. e.	Precipitation (metal hydroxide) Precipitation (metal sulfide) Precipitation (Aldrich Method) Electrowinning Ion Exchange (selective)

Source: Arthur D. Little, Inc.

- (a) Paint wastes contain a large fraction of diverse organic compounds which in themselves can classify the waste as hazardous and thus need to be addressed:
- (b) The quantity and character of the hazardous inorganic components in these wastes are not fixed but are highly dependent on the paint color being disposed; and
- (c) It is likely that, in some instances, the organic phase will constitute a large fraction of the paint waste.

Of the options listed in Table 4-4 it appears that at the first level, incineration or thermal treatment would be the appropriate first step due to its applicability to a wide variety of organic species. In this instance, it both separates the organic and inorganic fractions and converts the organic to a non hazardous state (eliminating the need for a second level treatment of the organic portion).

The approach that would be appropriate for the second level for the inorganics is highly dependent on the resultant composition of the ash. In the case where the ash is not classified as hazardous via the EPA TCLP, then no further treatment is needed prior to disposal. If, however, it is classified as hazardous, then attention needs to be focused on the particular metals present. In this case, the three approaches shown under Level II-1 may be appropriate. These are solubilization of the hazardous components and thermal treatment with or without additives (assuming all the organics have been removed). Vitrification of the ash after thermal treatment is a more stringent approach to fixation and is likely to be more universal (applicable to a variety of elements present in the waste). However, an approach which simultaneously treats the organic phase, separates out valuable metals and produces a vitrified non-hazardous residue would be most desirable.

At this stage, it did not seem appropriate to conduct any testing for the options listed under the third level. This is because the processes listed involve concentrating metals from leaching solutions and these processes have been amply studied.

4.3 Overview of the Laboratory Test Program

The applicability of combustion (incineration) or high temperature thermal treatment for destruction of the hazardous components of organic wastes has been shown to be practicable; hazardous waste incineration of organics is presently done and new methods for the high temperature thermal treatment or organics are always being evaluated. Therefore, the objective of the laboratory tests in this program was to focus on the affect of thermal treatment on the inorganic residues (pigments) and their potential hazardous classification by the proposed EPA inorganic TCLP. Based on the results of the TCLP tests of the ash samples, two samples were chosen for subsequent testing via glassification and/or possible recovery routes.

An overview of the approach used in the laboratory program is given in Figure 4-1. A summary of the tests conducted in the program is given in Table 4-5.

TABLE 4-5 SUMMARY OF LABORATORY PROGRAM

	Sample									
Type of Test	1.	2	3	4	5	6	7	8	9	10
Weight Loss/air dry	x	×,	x	x	x	x		x	x	x
Weight Loss/thermal treatment	x	x	x	x	x	x	x	x	x	x
Thermal Treatment:										
Fast 700°C	0	0	0	0						
Solids characteriza- tion	x	x	x	x						
TCLP of solids	x		x	x						
Ramp 700°C	0	0	0				0			
Solids characteriza- tion	x	×	x				x			
TCLP of solids	X		x				x			
Fast 1000°C	0	0	0	0	o	0		0	0	0
Sclids characteriza- tion	x	x	x	x	x	x		x	x	х
TCLP of solids	x		x	X.		x		x		x
Ramp 1000°C	0	0	0		0	0	0			
Solids characteriza- tion	x	x	x		x	x	x			
TCLP of solids	×		x			x	x			
Fixed 1100°C	0		0							
TCLP of solids	x	:	x							
Fixed 1450°C	0		0							
TCLP of solids	x		x							
<u>Glassification</u>										
Phosphate 800°C	0		0							
TCLP of solids	X		x							
Silicate 1100°C	0		0							
TCLP of solids	x		x							
Glassification and										
Recovery:										
Silicate & Carbon 1450°C	0	(0							
Solids characteriza- tion	x	;	x							
TCLP of solids	x		x							

Sample numbers represent the following:

See Section 5.0 (Laboratory Test Procedures) for explanation of tests.

Source: Arthur D. Little, Inc.

Arthur D. Little, Inc.

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¹⁾ epoxy primer

²⁾ CARC: 383-Green 3) CARC: Forest Green

⁴⁾ CARC: 383-Sand

⁶⁾ CARC: 383-Black

⁷⁾ Spray Booth Filter

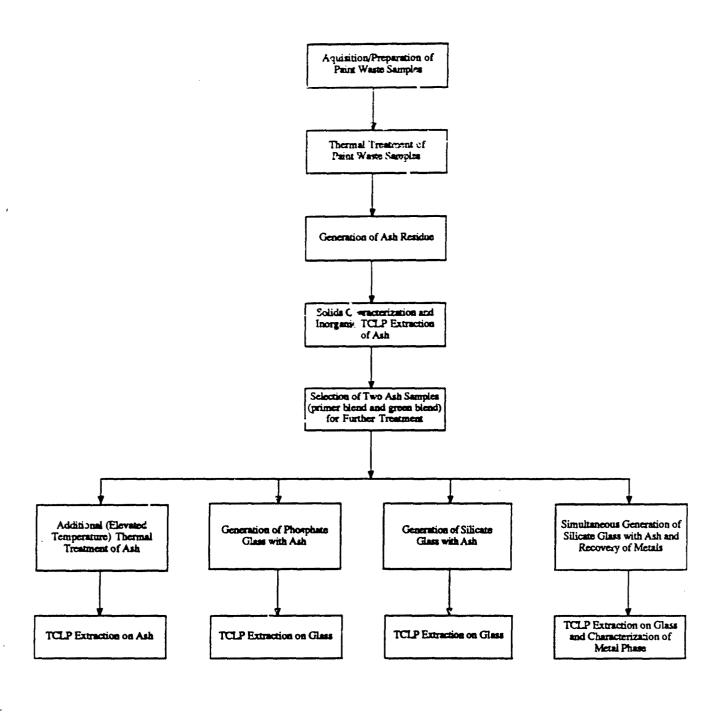
⁵⁾ CARC: 383-Brown 6) CARC: 383-8) Seafoam Green (epoxy/polyamide)

⁹⁾ Gray (epoxy/polyamide) 10) White (epoxy/polyamide)

o Represents test run.

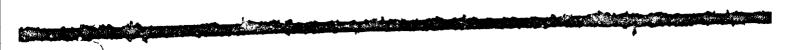
x Represents chemical analysis data.

Figure 4-1
Summary of Laboratory Approach



Source: Arthur D. Little, Inc.

Arthur D. Little, Inc.



5.0 LABORATORY TEST PROCEDURES

5.1 Sample Acquisition

Paint samples were, in general, supplied by the LEAD personnel and consisted of the following materials:

- · Epoxy Primer,
- Chemical Agent Resistant Coating (CARC) Paints [383-Green, Forest Green, 383-Sand, 383-Brown, and 383-Black],
- · Epoxy-polyamide Paints [Green, White and Gray], and
- · Spent Spray Booth Filter.

The epoxy primer sample was manufactured by Deft Chemical Coatings and consisted of two components (labelled A, a pigment compound, and B, a clear compound). It was labelled as primer coating, epoxy, water reducible, lead and chromate free adhering to MIL-P-53030.

The CARC paints all consisted of single component polyurethane paints. The 383-Green and 383-Brown colors were manufactured by the Midland Division of Dexter Corporation. They were labelled as Microthane and adhering to MIL-C-53039. The 383-Black and Forest Green colors were manufactured by Pratt and Lambert and were labelled as aliphatic polyurethane single component and as adhering to MIL-C-53039. The 383-Sand color was the only material received directly from the manufacturer, Hentzen Coatings, Incorporated. It was labelled Zenthane, polyurethane paint, one component, adhering to MIL-C-53039.

The epoxy-polyamide paints were two component materials manufactured by the Everseal Manufacturing Company. The white color was labelled as adhering to color specification C-17875 and had an Everseal product number 8-EP-611. The gray color was labelled Everseal product numbers of 8-EP-611 and Epoxy 26307. The green color was labelled as seafoam green, lead and chromate free, with Everseal product numbers of 8-EP-611 and Epoxy 24533.

The spent spray booth filter was received from LEAD on the 5th of May, 1987, and was collected immediately prior to that date. The sample was received dry at our laboratories.

5.2 Preparation of Samples

All the paint samples were prepared (in our laboratories) according to the paint manufacturer recommended procedures and allowed to air cure for a period of three days prior to measuring weight loss. An additional period of at least one week elapsed prior to performing any of the thermal treatment tests. The spent spray booth filters were received dry from LEAD and thermally treated (combusted) directly.

The green mix sample tested via the EPA TCLP was generated by mixing equal weights of the 383-Green and Forest Green solid residue products generated for these paints under the same thermal conditions. A Brown/Black (CARC) mix sample and a White/Gray (epoxy-polyamide) mix sample were similarly prepared.

The green mix sample used in the elevated temperature and additive tests was generated by blending approximately 40 to 50 gms each of eleven ash materials generated under the various thermal treatment (combustion) tests for the 383-Green and Forest Green materials. This mix was lightly ground using a mortar and pestle, screened thru a 35 mesh sieve, and then exposed to 1100°C in air for one hour (Footnote 1) prior to performing any additional tests.

The primer mix sample used in the elevated temperature and additive tests was generated by blending 80 to 150 gms each of seven ash materials generated in the various thermal treatment (combustion) tests with the epoxy primer material. This mix was then similarly sieved (35 mesh) and exposed to 1100°C in air for one hour (Footnote 1) prior to performing any additional tests.

5.3 Thermal Treatment (Combustion) Tests

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The conditions used in the small-scale thermal treatment (combustion) tests were chosen from both data on conditions expected in a commercial rotary kiln incinerator (currently the most commonly found manner of hazardous waste incineration) and the type of test equipment available at Arthur D. Little, Inc. The temperature range expected in a rotary kiln ranged from a low value of 700°C (1300°F), a medium value of 900°C (1650°F) to a high value of about 1200°C (2200°F). Heating rates range from a low of 0.1 deg/sec, a medium value of 5 deg/sec to a high value of 20 deg/sec.

The matrix of temperatures utilized in this program involved the following test conditions:

Condition (°C)	Heat rate (deg/sec)	Temperature (°C/°F)
Ramp 700	0.2 to 0.3	700/1300
Ramp 1000	0.25 to 0.5	1000/1800
Fast 700	3.3 to 5	700/1300
Fast 1000	5 to 7	1000/1800

A Thermcraft Cup Furnace Model 3.75-5-A-1183 was used for these experiments. This furnace has a 1 liter cup inserted into an oven which is preset at the desired temperature. The experiments were performed in two different modes.

Footnote 1: Heating was performed in order to generate starting material which would represent the most hazardous situation (via TCLP) of any conditions tested (see Section 6.0 for more discussion).

In the low heating rate mode, paint samples (5 to 20 gms) were inserted into the cup with the oven set at a low temperature (less than 100°C). The oven was then turned up to the final desired temperature (700 or 1000°C) and allowed to reach the final temperature, a process which typically took from 30 to 60 minutes. This translated to a heating rate in the range of 0.2 to 0.3 degree per second for 700°C tests and 0.25 to 0.5 degree per second for the 1000°C tests.

In the high heating rate mode, paint samples (5 to 15 gms) were inserted into the cup which was held at the final desired temperature. The cup was accessible so that samples were rapidly lowered (held by a platinum wire) into the cup and combustion/thermal decomposition occurred within a short period of time (usually complete within 2 minutes but varies with the size of sample and temperature). The samples were immediately removed from the cup after a 2 to 3 minute period. For samples which combust and reach final temperature within 2 to 3 minutes at 700°C, the heating rate is 3.3 to 5 deg/sec. Similarly, for a final temperature of 1000°C, the heating rate range is 5 to 7 deg/sec.

These experiments were repeated several times under identical conditions using the same sample to generate sufficient quantities of ash for subsequent analyses, extraction and treatment testing. The ash residues from these experiments (under identical conditions) was then composited.

In some cases, a further composite was prepared prior to performing subsequent tests. In the case of TCLP procedure, an equal mass composite of the 383-Green and of the Forest Green CARC paints was prepared and the mixture subjected to the extraction. Similarly, mixtures were prepared of the Gray and White colors of the epoxy/polyamine paints, and of the 383-Brown and 383-Black CARC paints for the TCLP analysis. These mixtures were prepared as such since the pigments present were similar in chemical components.

5.4 Elevated Temperature Tests

5.4.1 Without Additive(s)

The elevated temperature tests were performed in an electrically heated silicon carbide globar furnace. All these tests were conducted with the furnace set at the particular temperature and the material exposed for varying lengths of time. The fixed temperature exposure of the primer blend and the green blend ash samples were made by adding approximately 50 gms of the sample to a platinum crucible and heating the sample at the desired temperature (1100 or 1450°C) for a period of between 30 to 60 minutes for samples without additives.

5.4.2 With Additive(s)

The additive tests were performed by blending various amounts of additives with the primer blend or the green blend ash samples. The additives utilized included: sodium carbonate (Mallinkrodt Reagent Grade), carbon black

(Cabot MT-NS), Ferro[TM] unleaded phosphate frit (Ferro Corporation AL-59-2), Ferro[TM] leaded phosphate frit (Ferro Corporation AL-28-2), and a frit of sodium oxide and silica [25/75] prepared by mixing 42.5 gm of sodium carbonate and 75 gm of silica which was heated to 1200°C for 70 minutes with the resulting glass frit being cooled and ground to pass a 35 mesh sieve. The additive mixtures were placed in alumina crucibles and exposed to various temperatures and times (800 or 1100°C for 30 to 60 minutes). In recovery tests, mixtures (detailed in Section 6.0) of sodium oxide/silica frit, sodium carbonate, carbon black and the ash tested were placed in an alumina crucible. A 1/4 inch graphite disk was placed on top of the crucible (and a blanket of fiberfrax insulation was placed on top of the disk) to reduce the presence of oxygen in the crucible. The mixtures were exposed to 1450°C for 30 to 60 minutes.

5.5 EPA Extraction Procedure (TCLP)

Samples of the various inorganic residues were subjected to the inorganic portion of the TCLP procedure as described in the Federal Register, Vol 51, No. 114 (Friday, June 13, 1986) page 21685 with a modification of the sample mass subjected to the test. Since the laboratory experiments were designed on a small scale, instead of the customary 75 gms of the solid phase sample (used in step 7.0 of the procedure) a smaller sample was utilized.

A summary of this test procedure is given below:

- a) A representative sample of ash was weighed (20 to 50 gms) [Step 7.1].
- b) A small subsample of the waste (5 gms) was transferred to a 500 ml beaker, 96.5 ml of distilled water added, stirred for 5 min using a magnetic stirrer and the pH recorded. If the pH was <5 extraction solution 1 (pH of extraction solution is 4.93) was used. If the pH was >5, 3.5 ml of 1.0N HCl was added, the slurry heated to 50 degrees C for 10 minutes and cooled prior to measurement of pH. If the pH was <5 extraction solution 1 was used and if it is >5 extraction solution 2 (pH of extraction solution is 2.88) was used [Step 7.12].
- c) To the extractor (e.g a 2 liter bottle) 20 to 50 gms of the waste was placed and 20 times its weight of the proper extraction solution determined in step b above was added.
- d) The extractor bottle was placed into a rotary extractor device capable of rotating at about 30 rpm for 18 hours. Room temperature was used.
- e) The slurry was then filtered through a 0.6-0.8 micron borosilicate glass fiber (Whatman GF/F) filter (without any prefilter) which had been previously washed with 1.0 N HCl followed by three consecutive rinses of 750 ml of distilled water.

f) The filtered liquid phase of this slurry is defined as the TCLP extract. The extract was preserved using 0.5% of concentrated nitric acid (Baker Instra Analyzed).

Based on the total analysis results on the starting inorganic residue samples, it was deemed that there was no need to analyze for Hg, As and Se in the extracts. The analysis method used was inductively coupled argon plasma (ICAP) spectroscopy which is capable of analyzing the selected metals simultaneously. Samples analyzed by Barringer Magenta were acid digested prior to analysis [as required in Step 7.16] The regulatory levels for the elements listed in mg per liter of leachate are: As (5.0 mg/l), Ba (100 mg/l), Cr (5 mg/l), Pb (5 mg/l), Hg (0.2 mg/l), Se (1.0 mg/l), and Ag (5.0 mg/l).

5.6 Chemical Analysis Procedures

5.6.1 Dissolution of Solids

The analysis of the inorganic residue (ash) of the combustion experiments was performed to determine the composition of the ash for major components as well as the metals of concern in the TCLP procedure. The major components were expected to be those related to the pigments (shown in Table 4-2)) and include Ti, Fe, Zn, Cr, Mg, and Si. The metals of concern in the TCLP procedure were Ba and Cr. Minor amounts of the other TCLP metals were expected from use of paints which are not large volume and from impurities present in the pigments of the large volume colors.

The analysis procedure for the solid ash samples utilized a variety of fusion or digestion procedures depending on the analyte of interest. These were:

- Hydroflouric/nitric/perchloric digestion for ICAP analysis,
- Nitric/perchloric (not to dryness) for As and Se,
- · Nitric/Sulfuric/Permanganate/Persulfate for Hg, and
- Sodium carbonate fusion (make up in 10 % nitric) for B and Si.

These procedures were performed by Barringer Magenta Ltd., Rexdale, Ontario.

5.6.2 ICAP Analysis of Solution and Digests

I

ICAP analysis (performed by Barringer Magenta, Ltd.) utilized an Applied Research Laboratories spectrometer. The analysis is performed by aspirating the undiluted sample which provides emission data for trace and major components (up to 1000 ppm). A computer-based program which corrects for inter-element spectral interferences is used to quantify the trace elements. The major species (>100 ppm) are quantitated by diluting the original sample and reanalyzing it. These

reanalyzed values are used as input concentrations for major species in the interelement correction program.

The following elements were analyzed simultaneously for liquid samples: Ag, Al, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, P, Si, Sr, Th, Ti, V, Zn and Zr. In the case where solid samples that have been digested with mixtures containing HF an additional fusion was performed for B or Si due to their loss in the digestion procedure by formation of volatile fluorides.

ICAP analyses were also performed at Arthur D. Little, Inc. using a Thermo Jarrell Ash Plasma 300 dual channel sequential analyzer. This instrument was used to analyzed Ba and Cr in extracts for comparison purposes. The wavelength used to monitor these elements were 493.4 and 233.5 nm for Ba and 205.5 nm for Cr. Standardization was made using individual standards prepared from Spex Industries solutions.

5.6.3 Atomic Absorption (AA) Analysis of Solutions

Analysis of ash sample digests for As, Hg, Pb and Se were performed by Barringer Magenta, Ltd. following methods specified in "Methods for Chemical Analysis of Water and Wastes" (1983 Revision). Arsenic and selenium were analyzed using hydride generation (via reduction with sodium borohydride) and heated quartz tube atomic absorption detection on a Varian Model AA5 (Methods 206.3 [As] and 270.3 [Se]). Mercury was analyzed using cold vapor generation (using stannous chloride) and a Laboratory Data Control LDC Model 1205 Mercury Monitor (Method 245.2 and 245.5). Lead was determined using flame atomic absorption (Method 239.1) using a Varian Model AA5.

5.6.4 Solid State Characterization

A Phillips X-ray unit (at Arthur D. Little) was used to obtain X-ray diffraction (XRD) patterns on each of the samples prepared at elevated temperatures. The primary intent of the XRD analysis was to determine if the heated materials contained amorphous and/or crystalline phases. In each of these diffraction analyses, settings of 40KV and 20 MA were used. The samples were scanned in a 2 theta range from 10 degrees to 55 degrees copper target.

Total organic carbon was determined using a LECO Model 521-000 Carbon Analyzer following the manufacturer procedures.

Elemental analysis was also determined (at Arthur D. Little) on some of the elevated temperature samples using a Cambridge STEREOSCAN, scanning electron microscope (SEN) with a KEVEX-EDX 7500 microanalysis attachment (x-ray fluorescence).

5.7 Quality Assurance/Quality Control

Following discussions with U.S. Army Toxic and Hazardous Materials Agency (USATHAMA), this research program was deemed not to require the use of USATHAMA Certified Procedures. Thus, the procedures used did not adhere to those specified in the "Sampling and Chemical Analysis

Program for the U.S. Army and Toxic Hazardous Materials Agency (Dec 1985) Guidelines." The quality control samples listed below were designed specifically for this task. A variety of quality control (QC) samples and procedures were utilized throughout the laboratory testing and analysis process. These included:

- · Blanks generated from digestion of solids,
- · Duplicate analyses of solid digests,
- · Both known and blind solid standards (NBS),
- . Both known and blind liquid standards (NBS and EPA),
- · Internally generated pool standard solutions,
- · TCLP extract blanks,
- · Replicate analyses of sclution samples,
- · Replicate analyses of TCLP extracts,
- · Spikes of TCLP extracts, and
- · Interlab comparison for Ba and Cr analyses of solutions.

These QC samples were either utilized by Barringer Magenta Ltd. in the conduct of their analyses or inserted blindly into the sample batches by Arthur D. Little, Inc. personnel. A summary of the number and type of QC samples used in the program is given in Table 5-1. All of the results are provided in Appendix C and are discussed in section 6.4.

TABLE 5-1

SUMMARY OF NUMBER AND TYPE OF QUALITY CONTROL SAMPLES

OC Sample	Barringer <u>Magenta</u>	<u>ADL</u>	<u>Total</u>
Solids Analyses [a]:			
Blanks Replicates Standards [d]	6 11 7	1 [c]	6 12 8
Internal Standards [g]	6	-	6
Liquid Analyses [b]:			
Blanks	3	6 [c,f]	9
Replicates	_. 5	3 [c,f]	8 5
Standards [e]	2	3 [c,e]	5
Internal Standards	4	•	4
Spikes [h]	8 [h]	•	8
Intralab Comparison [i]	•	13	13

a. Total number of solid samples for data was 23, excluding QC samples.

Source: Arthur D. Little, Inc.

b. Total number of liquid samples for data was 28, excluding QC samples.

c. Blind samples

d. NBS standards (1633a or 1645)

e. NBS (1643a) or EPA (Mineral) standards.

f. TCLP extracts.

g. Labelled by Baringer Magenta Ltd. as control, pool, sy-1 or sy-2.

h. TCLP extracts at two concentration levels.

i. Ba and Cr analyses via ICAP.

6.0 LABORATORY TEST RESULTS

6.1 Thermal Treatment (Combustion) Tests

6.1.1 Solid Residue Composition

A summary of the weight loss observed for all the samples tested is given in Table A-1 in Appendix A. In general, all the paint samples tested had an amount of material remaining after air drying between 71 to 88%. After thermal treatment (combustion) of the dried residues at temperatures ranging between 700 and 1000°C, the amount of ash generated was 36 to 53% of the original dried weight of the CARC paints and between 33 and 55% of the epoxy/polyamide paint. The spent spray booth filter generated ash at approximately 56% of its original weight. These data indicate that if undried paint materials were subjected to thermal treatment to remove the organic components, the overall residue would be between 25 and 57% of the original weight (assuming no other solvents were mixed in with the waste).

A summary of the major components found in the various types of paint samples tested in shown in Figure 6-1. All of the samples contained silicon and iron while the other elements were present in varying concentration in the different samples. Individual ash characteristics are discussed below:

Epoxy primer - The major components of the epoxy primer ash (Table A-2) were: Fe (15%), Ba (12%), Zn (11%), Si (8%), P (3.5%), Mg (2.8%), and Al (1.8%). The remaining elements analyzed were at lower levels. Calcium was found at 0.9%. The concentration of Ag, As, Se, and Hg were very low. The ash products generated at 700°C had residual total organic carbon (TOC) of about 3 to 5% which was reduced to 0.6 to 2.4% in the 1000°C tests. No other great differences were observed from exposure to the different heating rates or temperatures.

383 Green and Forest Green (CARC) - The major components in the ashes from these paints (Tables A-3 and A-4) were: Cr (17-20%), Si (18-20%), Mg (7%), Fe (3-5%), Zn (2-3%), Co (1-2%). The remainder were present at lower levels. Al and Ca were found at 0.4% and 0.1%, respectively. Trace amounts of As, Se, Hg or Ag were found. Not much impact on the TOC levels (0.2%) was observed between 700 and 1000°C. Little impact of the various heating conditions was observed on the ash composition.

383 Sand (CARC)- The major components of the ash were (Table A-5): Si (28%), Ti (12%), Cr (3.7%), Fe (3.4%), Al (1%) with the remainder of the elements analyzed at lower levels. Calcium was found at 0.1%. Trace amounts of Ag, As, Se, Hg and Pb were found. A reduction of TOC from 0.64 to 0.16% was observed in the 700 to 1000°C ashes. No major changes were observed due to heating under the various conditions.

383 Brown (CARC)- The major components of the ash generated from this paint were (Table A-6): Si (24.5%), Cr (12%), Fe (9.3%), Mg (7%). The remainder of the elements, e.g. Al (0.6%) and Ca (0.1%) being at low levels. Trace amounts of Ag, As, Se, and Hg were found. The residual TOC ranged from 0.08 to 0.44 in the 1000°C ashes.

Source: Arthur D. Little, Inc.

383 Black (CARC) - The major components of the ash were (Table A-7): Si (27%), Fe (14%), Mg (10%). The remainder were at lower concentrations (e.g., Al at 0.6%). Trace levels of As, Se, Ag and Hg were present. The remaining TOC at 1000°C was relatively low (less than 0.02 to 0.08%).

Spent Spray Booth Filter - The major components of the ash generated from this sample was (Table A-8): Si (20%), Cr (13%), Fe (7%), Zn (4.7%), Ba (3%), Mg (1.8%), Co (0.9-3%), and Al (0.9-1.4%) with the remainder lower. Greater variability was observed in the two samples tested under the different thermal conditions (700 and 1000°C) however, this variability was probably due to the inhomogeneity of the samples. The distribution of paint on the spent filter was found to be very inhomogeneous. Trace levels of Ag, As, Se and Hg were found. The residual TOC ranged from 2.7% at 700°C to 0.2% at 1000°3.

The major components in this sample suggest that the paints being utilized in the spray booth were probably the epoxy primer (which contributes Ba and Zn) and the various green pigments (which contribute Cr and Co).

Epoxy/Polyamide paints - The major components of the ashes generated from these paints were (Table A-9):

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Green - Si (28%), Ti (22%), Cr (1.5%), Fe (1.5%)
Gray - Si (28%), Ti (25%)
White - Ti (44%), Si (8.5%), Al (3.3%)
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In all these ashes, trace levels of Ag, As, Se, Pb and Hg were found. The residual TOC ranged from 0.6 to 1.7% after thermal treatment at 1000°C.

For all the 2sh samples analyzed, the likely anion associated with all the catonic elements analyzed is oxide (see Tables 4-2 and 4-3) which provides the remainder of the mass balance.

6.1.2 TCLP Extracts Data

All of the extract chemical data is given in Appendix A. A summary for the TCLP regulatory metals is given in Table 6-1. None of the ashes generated in the temperature range of 700 to 1000°C were classified as hazardous. The epoxy polyamide paints generate ashes which had lower TCLP concentration than the CARC paints or epoxy primer materials. The elements that appear at the highest concentrations in the extracts are Ba (for the epoxy primer) and Cr (for the green mix). Temperature also has an impact on the extract concentration of barium which is 43% of the regulatory criteria in the fast 1000°C tests. The green mix generated a concentration of chromium which is 31% of the criteria in the ramp 1000°C test. None of the other metals of regulatory importance generated concentrations of concern. The higher Ba and Cr extractability was confirmed by tests done on the ash at higher temperatures as discussed in the following sections.

TABLE 6-1

Concentrations of Regulatory Metals in TCLP Extracts
For Ashes of Various Paints

SAMPLE\TEST	PLENTEST Thermal			Concentration			[a]				
,	Cond	ition	Ag	Ba	Cd	Cr	Ni	Pb	As	Se	Hg
Epoxy Primer	Fast Ramp	700 700 1000 1000	BDL BDL		BDL 0.05	BDL	0.2 0.23 BDL 0.16	BDL BDL	400 400	3.5	2 2
CARC (Green Mix)	Ramp	700 700 1000 1000	BDL BDL	0.18 0.13 0.27 0.33	BDL BDL	0.12 0.11 1.56 1.16	BDL BDL	BDL BDL BDL BDL	60 50 	4 2 	
CARC (Sand)	Fast Fast	700 1000	BDL BDL	0.20 0.21		BDL 0.19	BDL BDL	BDL BDL	70 130	BDL 1	0.3 0.3
CARC (Brown/Black)	•	1000 1000		0.56 0.31		0.35 BDL		0.2 BDL	100		0.3
Spray Filter	•	700 1000		0.99 0.26	0.08 0.09				••		
Epoxy/Amide (Green)	Fast	1000	BDL	0.23	BDI.	BDL	BDL	BDL	0.45	BDL	2.1
Epoxy/Amide (White)	Fast	1000	BDL	0.25	BDL	BDL	80.0	BDL	0.3	0.00	0.35
Regulatory Leve	el(ppm	ı)[a]	5	100	1	5	15	5	5000	1000	200

Note a: All concentrations are in ppm except for As, Se and Hg which are in ppb. Data may not be identical to that in other tables due to rounding. BDL - Below detection limit. The data for As, S. and Hg were calculated from concentrations in the solid phase.

6.2 Elevated Temperature Tests (Without Additives)

6.2.1 Solid Residue Properties

As would be expected from their chemical compositions (Table 6-2), the epoxy primer pigment behaved very different from the green mixture pigment. The epoxy primer contains appreciably more phosphate and iron oxide and much less chromiur oxide than does the green mix. The higher Cr and lower Fe content of the green ash results in a higher melting material. The green mix heated to 1450°C gave only a lightly fused non-volctile mass whereas heating of the epoxy primer to 1100°C caused the material to melt. Furthermore, X-ray diffraction showed the green mix to remain crystalline whereas the epoxy primer was converted to an amorphous or glassy material (Table 6-3). Generally speaking the higher the temperature of which a glass softens the more durable and less leachable it is. Compound such as aluminum oxide and chromium oxide tend to raise the softening temperature of a glass whereas compounds such as iron oxide, alkalies and phosphates reduce the softening temperature (act as fluxes). In the case of the green mix a small amount of liquid phase was generated at the elevated temperature as evident from the X-ray results of Table 6-3 and from the fact that the material was found to only lightly sinter at 1450°C and remained as a powder when heated to 1100°C.

From these experiments it also became evident that in any high temperature process (i.e., above about 1100°C), zinc volatility will have to be considered and addressed. A zinc containing compound, as shown in Figure 6-2, was found to condense on some of the upper-outside cooler regions of the platinum crucible (in the 1450°C test). These vapors condensed and formed a whisker-like material which was confirmed by x-ray diffraction patterns to be zinc oxide. While the SEM-KEVEX (x-ray fluorescence) showed a depletion of zinc from the starting material (Figure B-4, Appendix B).

6.2.2 TCLP Extract Data

Epoxy primer ash - The TCLP extract data for epoxy primer ash subjected to elevated temperatures (Table 6-4) indicate affects in two opposite directions with regard to leachability of particular elements. The most important result observed is the increasing leachability of barium (a TCLP important element) with higher temperatures. This trend is evident from 700°C to 1100°C with the latter having concentrations which begin to exceed the 100 ppm criteria. (Note that the fixed 1450°C ash product was leached using leaching fluid #2 which indicates a higher alkaline content than the fixed 1190°C product but the higher barium level observed in the leachate is probably a result of both further decomposition as well as the lower pH of the extracting fluid). Decomposition of a barium sulfate (found by XRD), presumably to barium oxide, explains the observed data. Other elements (Table A-26, Appendix A) which also show increased leachability going from 1000 to 1100°C but to a much lesser extent are Fe, K and P. Similarly, the increase P levels may be the result of decomposition of zinc phosphate.

TABLE 6-2

COMPARISON OF STARTING ASH TO FINAL GLASS PRODUCT [a]

Component	Primer Ash (%)	Primer Glass (%)	Change (%)	Green Ash [b] (%)	Green Glass (%)	Change (%)
Al	1.8	1.3	-28	0.5	NP	-
Ва	12	13.7	14	NP	NP	-
Ca	0.9	1.0	11	0.2	0.6	200
Co	NP	NP	-	1 5	0.1	-93
Cr	NP	NP	-	18.5	0.9	- 95
Cu	NP	NP	-	NP	NP	-
Fa	14.7	5.0	-66	3.7	0.2	-95
K	0.77	1.3	69	NP	NP	-
Mg	2.81	0.3	-89	7.2	3.2	-56
Иa	0.17	1.5	782	0.5	1.4	180
P	3.39	0.3	-90	NP	NP	-
Si	7.52	29.5	294	19.5	40.8	109
S	[c]	1.6	[c]	NP	NP	-
Ti	NP	NP	-	0.9	1.5	67
Zn	10.8	1.2	-89	NP	NP	-

Note a: Chemical analysis performed using SEM/EDX. Tests run at 1450 degrees centrigrade with carbon, sodium carbonate and silica addition.

Initial composition has not been adjusted to reflect dilution by the additives which represent approximatly 50%.

Note b: Average composition for 383 Green and Forest Green ashes.

Note c: Sulfur was not analyzed in the initial ash but barium sulfate was determined to be present by XRD

NP - Not present as a major component (less than one percent)



TABLE 6-3

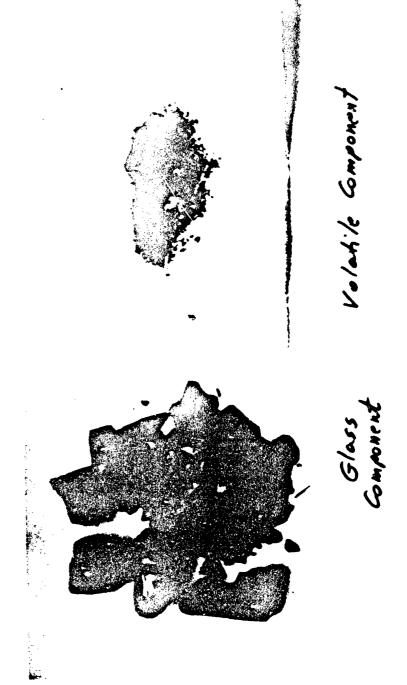
X-RAY DIFFRACTION ANALYSES OF SOLIDS GENERATED IN ADDITIVE TESTS

Sample	Number	X-Ray Diffraction Results		
Primer Blend:				
700°C	9	crystalline		
1000°C	10	crystalline		
1100°C	100	mostly amorphous		
1450°C (glass phase)	105	amorphous [a]		
1450°C (volatilized product)/ 1450°C	103	crystalline (ZnO)		
phosphate additive/800°C	109	amorphous		
sodium carbonate/1100°C	107	mixture [b]		
Green Blend:				
700°C	24	crystalline		
1100°C	101	crystalline		
1450°C	106	crystalline		
leaded phosphate/800°C	110	amorphous some crystalline		
unleaded phosphate/800°C	111	amorphous some crystalline		
sodium silicate/1100°C	112	mixture		

Note a: no crystalline peak evident.

Note b: both amorphous and crystalline pattern.

FIGURE 6-2. EPOXY PRIMER ASH HEATED 1450°C FOR 30 MINUTES



6-8

TABLE 6-4

Concentrations of Regulatory Metals in TCLP Extracts
For Treated Ashes

Sample [b]	[c]	Ag	Ba	Cd	Cr	Ni	Pb	Fluid No.[d]
Primer		BDL BDL BDL	0.31 0.38 25.8	BDL BDL 0.05	BDL BDL BDL	0.2 0.23 BDL	BDL BDL BDL	1
Primer Mix 1	1100 -H 1450 -H 1100 -Sil 800 -Phos 1450 -C+S	BDL BDL BDL	775 678 300	BDL 0.06 0.02	BDL 0.35 0.15	0.41 0.26 0.08		1 2 2 2 1
Green Mix 1	700 -R 700 -F 1000 -R 1000 -F	BDL BDL	0.13 0.27	BDL BDL	0.11 1.56	BDL	BDL BDL	1 1 1
Green Mix 2	1100 -H 1100 -dup 1450 -H 1100 -Sil 800 -Phos 1450 -C+S	BDL BDL BDL BDL	0.64 1.27 4.42 3.74	BDL BDL BDL BDL	3.63 0.15 13.8 9.27	BDL BDL BDL BDL		1 1 1 1 1
Regulatory Leve	el (ppm)	5	100	1	5	15	5	

Note a: All concentrations are in ppm.

Note b: Primer mix 1 consists of a blend of all ashes generated in previous primer tests.

Green mix 1 consists of equal mass mixture of 383-Green and Forest Green ash materials generated under the same condition Green mix 2 consists of a blend of all ashes generated in the previous 383-Green and Forest Green tests.

Note c: Abbreviations used are as follows-

R- ramp; F- Fast; H- Held Fixed; dup- duplicate
Sil - sodium carbonate and sodium oxide/silica frit
additives (the latter for green mix 2 only).
Phos - unleaded Ferro(TM) phosphate frit additive

C+S - carbon, sodium carbonate, and sodium oxide/silica frit additives

Note d: Fluid 1 is a sodium acetate buffer at pH 4.9 Fluid 2 is an acetic acid solution at pF .88

Source: Arthur D. Little, Inc.

Green ash - The most important affect of temperature observed for the green pigments ash was the increased leachability of chromium (concentration of 3.7 ppm) as the temperature was increased to the fixed 1100°C (Table 6-4). Further raising of the temperature to the fixed 1450°C caused the leachability to decrease (concentration of 0.15 ppm). These results can be explained by conversion of the chromic(III) oxide to a more soluble chromium(IV) or chromium(VI) oxide form aided by temperature and oxygen at temperatures between 700 and 1100°C. As the temperature is further raised to 1450°C, loss of oxygen from these products can occur to reduce the chromium back to less leachable forms. In addition, the residual TOC in the samples may have participated in this reaction. Similarly, for iron, increased leachability was observed (to 1.5 ppm) at the fixed 1450°C test. This result can be explained by conversion of the iron(III) oxide to a more soluble mixed iron(II) and iron(III) oxide via loss of oxygen. The data shown in Table A-27, Appendix A also indicates that calcium, magnesium and silicon were involved in a reaction to produce a glass since a decrease in leachability of these elements was noted from the asn product. However, the product was only glassified to a small extent since the XRD results indicated that it was mostly crystalline.

6.3 Elevated Temperature Tests (With Additives)

6.3.1 Solid Residue Properties

The experiments using additives to the pigment ash mixes can be categorized into two types, oxidizing and reducing conditions. A summary table of the various conditions used is given in Table 6-5. It should be noted that in these experiments (principally the oxidizing) the glass additives/resultant glass composition was not optimized relative to leachability/durability but rather should be viewed as experiments to demonstrate that the ions of interest (i.e., barium, chromium) can be incorporated into a glass matrix.

It is believed that a durable glass of formulation/additive can be obtained which would decrease the leachability of these ions. Most likely a borosilicate based glass would provide the necessary degree of durability. To achieve such a formulation at least a several person-month effort would be required; this was considered to be outside the scope and budget of the present program.

From Table 6-2 it is readily apparent from comparing additive versus non-additive specimens at equivalent temperatures (under oxidizing conditions) that an additional degree of glassification (as evidenced by the presence of an amorphous phase) occurred in the epoxy primer material and that glassification of the green mix occurred. The X-ray diffraction results (Table 6-2) indicated that most but not all of the green pigment was glassified. Furthermore, from Table 6-4 it may be seen that merely adding sodium carbonate to flux the epoxy primer (1100-sil) did not produce sufficient glass to totally incorporate all of the crystalline phases into a glassy network at 1100°C. Similarly, the silicate and phosphate additives to the green ash (1100-sil and 800-phos) were not sufficient to incorporate all the crystalline phases.

TABLE 6-5
SUMMARY OF ADDITIVE TEST CONDITIONS

Sample	Additives	Temperature	Time (min)
Primer 1	Blend:		
109	Ferro unleaded phosphate frit (13.5g. Sodium Carbonate (1.5g) Primer Ash (15.0 g)) 800	30
107	Sodium Carbonate (3.0 g) Primer Ash (25.0 g)	1100	20
S-1	Sodium Carbonate (2.0 g) Primer Ash (20 g)	1000	20
S-2	Ammonium Phosphate (4.0 g) Primer Ash (20 g)	870	30
113	Sodium oxide/silica [25/75] (30.0 g) Carbon black (4.5 g) Sodium Carbonate (3.0 g) Primer Ash (30.0 g)	1200	30
120	Sodium oxide/silica [25/75] (40.0 g) Carbon black (7.0 g) Sodium Carbonate (2.5 g) Primer Ash (40.0 g)	1450	60
Green Bl	end:		
S-3	Sodium Carbonate (2.0 g) Green Ash (18.0 g)	1100	20
S-4	Sodium Carbonate (2.0 g) Ammonium Phosphate (3.9 g) Green Ash (18.0 g)	1100	20
110	Sodium Carbonate (1.5 g) Ferro leaded phophate frit (13.5 g) Green Ash (15.0 g)	800	30
111	Sodium Carbonate (2.5 g) Ferro unleaded phosphate frit (20.0 g Green Ash (20.0 g)	800	30
112	Sodium oxide/silica [25/75] (20.0 g) Sodium carbonate (2.5 g) Green Ash (20.0 g)	1100	30
116	Sodium oxide/silica [25/75] (40.0 g) Sodium carbonate (4.4 g) Carbon Black (2.5 g) Green Ash (40.0 g)	1450	30
118	Sodium oxide/silica [25/75] (40.0 g) Sodium carbonate (2.5 g) Carbon Black (7.0 g) Green Ash (40.0 g)	1450	60

Note: All tests were done under oxidizing conditions except those where carbon black was added which were done under reducing conditions.

In the experiments under a reducing condition for both the epoxy primer and the green mix samples, the amount of carbon used was in excess (by approximately 8%) of the stoichiometric amount needed to reduce all of the iron, chromium, phosphorus and zinc oxides available in each of the pigments. In Figure 6-3, the relative ease of reducing the various metal oxides is shown. The line representing reaction of carbon and oxygen to produce carbon monoxide intersects the Cr/Cr₂O₂ line at about 1200°C, the Zn/ZnO line at about 950°C, the Fe/FeO line at about 700°C and the CoCoO line at about 500°C. The intersection point represents the minimum temperature above which the reduced metallic state is favored over the metal oxide. From Figure 6-3, it may be seen that at a temperature of 1450°C the oxides of each of the above mentioned elements should be reduced to the metallic state. Furthermore, the phase diagram (Figure D-1, Appendix D) shows that at this temperature and the expected Cr to Fe ratio of the green ash, a liquid phase with about 70% Cr and 30% Fe would not occur due to the higher melting point (>1700°C). However, because of impurities such as phosphorous, the melting point of iron/phosphorus phase is lowered to below 1450°C (Figure D-2, Appendix D) for the primer ash and the metal phase did melt.

For both the epoxy primer and the green mix samples a metallic phase and a glass phases in these two tests were formed under the reducing conditions. The relative concentrations of important elements in the starting epoxy primer ash, glass and the metal product generated in the recovery test is shown in Figure 6-4. A similar comparison for the green CARC paint mixture ash is shown in Figure 6-5. The metallic phase consisted primarily of the metals of interest, namely iron and chromium, with other components (e.g. P, Co, Ba) present in smaller amounts (see Table 6-6 and Figures B-1 and B-2, Appendix B). Conversely, in the glass phases, these same elements were found to be depleted relative to the starting ash composition (Table 6-3 and Figures B-3 and B-4, Appendix B). Thus, chromium was removed from the glass product of the green ash leading, as expected, to a non-hazardous residue while a large amount of barium from the epoxy primer ash was incorporated into the glass phase.

6.3.2 TCLP Extract Data

A comparison of concentrations of important elements in TCLP extracts of starting and product materials obtained from the various glassification and recovery tests is given in Figure 6-6 for the epoxy primer and in Figure 6-7 for the green CARC mixture. The results are discussed below.

Epoxy primer and - The high barium concentrations in extracts of the Silicate-1100°C test (678 ppm, Table 6-4) indicate that addition of only sodium carbonate (approximately 11%) and using the existing silica in the ash was not sufficient to produce a non-hazardous residue. The lower concentration of barium (18.7 ppm) observed in the extract of the glass/metal product of the recovery 1450°C test (with 11% sodium carbonate and 33% silica added) indicates that the glass generated incorporated a great deal of barium and was not hazardous. In addition, the glass generated resulted in reduced leachability of

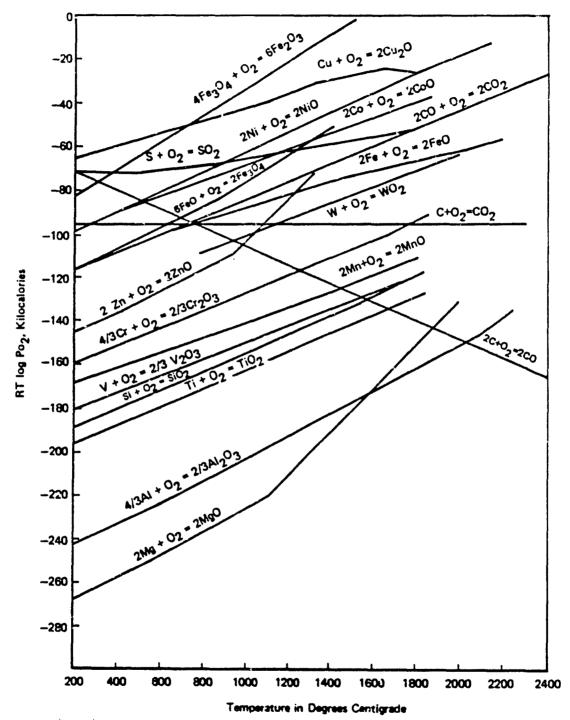


FIGURE 6-3: Relative Reducability of Various Metal Oxides

Source: H.J.T. Ellingham, J. Soc. Chem. Ind., 1944, Vol. 63, p. 127, Society of Chemical Industry.



Source: Arthur D. Little, Inc.

TABLE 6-6

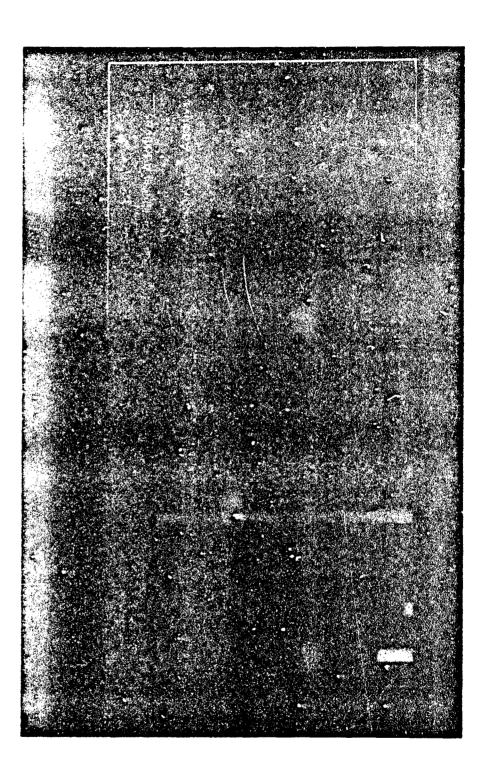
CHEMICAL COMPOSITION OF METAL PHASES GENERATED IN GLASSIFICATION AND RECOVERY TESTS [a]

Component	Metal From Primer Ash (%)	Metal From Green Ash (%)
		76 62
Cr	NP	76.43
Fe	81.14	16.44
P	7.68	NP
Ba	6.67	NP
	NP	5.77
Co	1.68	NP
Zn	NP	1.08
Ti	0.94	0.78
Si	• •	NP
Na	0.66	NP
S	0.45	
Al	0.24	0.03
Ca	0.35	0.21
K	0.19	NP
r Mg	NP	NP

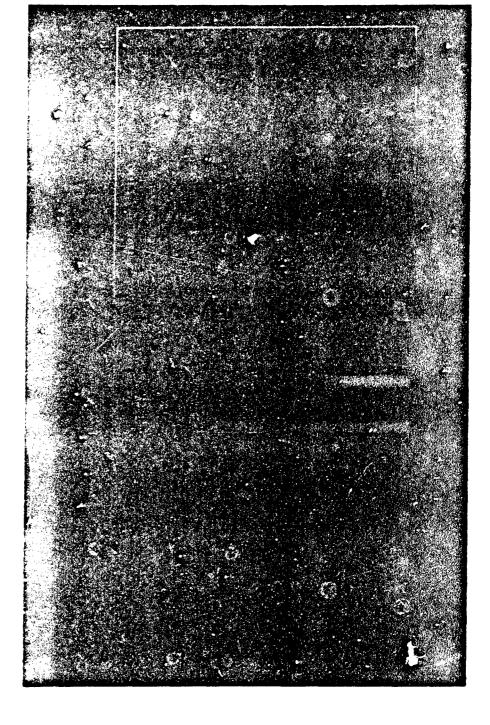
Note a: Tests performed at 1450 degrees centrigrade with carbon, sodium carbonate and silica addition.

Chemical composition determined using SEM/EDX.

NP = Not present as a major component (less than one percent)



Arthur D. Little, Inc.



IGURE 6-

boron, calcium, potassium, magnesium, manganese, strontium and zinc (Table A-27, Appendix A) as compared to the leachability for the residue without additives in the fixed 1100°C test. The metal phase generated in the recovery-1450°C test incorporated iron, phosphorus and possibly zinc. The phosphorus and zinc may have also partially volatilized in this experiment. Any residue of these elements in the ash phase appears to also been incorporated into the glass as evidenced by the lower leachability.

The high concentrations of iron (128 ppm) in the extracts of the epoxy primer (and the green ash) product of the recovery-1450°C test relative to other extracts in the program (0 to 8 ppm), as shown in Table A-26 (and A-27) in Appendix A, suggests that the iron is not associated with the glass matrix. These high dissolved concentrations are consistent with iron powder (exposed by grinding the glass/metal mixture) which was surface oxidized to produce a readily soluble iron(III) oxyhydroxide. This also indicates the presence of unseparated metal phase in the product extracted. This is very likely since no separation of the two phases was possible in these tests. The presence of metal in the glasses tested and the fact that neither the primer nor green glasses generated a hazardous TCLP extract (i.e. chromium concentration in the extract of the green ash was 0.18 ppm) indicates the ability to dispose of the impure glass phase or the metal phase (Cr/Fe) as a non-hazardous waste. It also suggests that at the relatively lower temperature (1450°C), the chromium metal did not alloy with the iron (since neither melted). In a situation where an alloy was generated (i.e. similar to stainless steel) the extractability would be expected to be lower. The metal phase generated from the primer ash (Fe/P) did melt but its durability is expected also to be lower than a Cr/Fe alloy.

Green ash - The higher chromium levels (13.8 ppm) found in the extracts of the silicate-1100°C product (where 11% sodium carbonate and 33% silica were added) relative to the fixed-1100°C ash coupled with the decrease in concentration of B, Ca, Mg, Si, Sr and V indicate that the silicate glass produced did not incorporate much of the chromium and it would be classified as a hazardous waste. It may be necessary to increase the silicate additives by a factor of 2 to 4 to incorporate all of the chromium into a glass under these conditions. The glass/metal product of the recovery-1450°C test resulted in a low chromium extractability (Table 6-4). This is onsistent with a conversion of chromium oxide to chromium metal thus redu ing the amount that needs to get incorporated into the glass structure. The beneficial effects of carbon thus appear to be:

- 1) Reduction of the amount of chromium oxide in the waste so that it can be glassified more easily,
- 2) Generation of a chromium rich metal phase which can potentially be recovered, and
- 3) Generation of a chromium/iron/cobalt alloy which is leach resistant and can be disposed of as a non-hazardous waste.

The glass product generated in the fixed 1450°C test also showed reduced chromium leachability consistent with either chemical conversion or incorporation into the glass. A glass phase was generated (as evidenced by the data shown in Table A-27 which shows reduced leachability of magnesium, silicon and calcium relative to the data for the fixed-1100°C test) but of poorer quality than that in the recovery-1450°C test (as evidenced by higher extract concentrations of magnesium, silicon and calcium in the former test). material did not melt in this test, the glass produced may have been a localized phenomenon around small particles and may have coated the particle sufficiently to reduce leachability. The high extract concentration in the fixed-1450°C test for iron is similar to the change in extractability observed in the recovery-1450°C test. This suggest that iron species may have reacted with the chromium, reducing the latter extractibility while increasing the former in a similar fashion to what was observed when the carbon reacted with the iron on the recovery-1450°C test. This increased leachability of iron may also have been simply a result of thermal decomposition of Fe₂O₂ toward more soluble FeO at these elevated temperatures.

Other elements which appear to have reduced leachability and thus are presumably incorporated into the glass are B, Ca, Mg and Sr (Table A-27, Appendix A) similar to that observed for the epoxy primer.

The cobalt concentration in the extract of the recovery-1450°C glass/metal product was increased greatly (to 8.5 ppm) above the level in other tests (0 to 0.5 ppm). This behavior tracks that of iron in these tests and is explainable in a similar fashion in that a leachable oxide coating was created on the fine particle surfaces during the grinding operation. It should be noted that in an actual disposal scenario, the glass would not be ground and would encapsulate any residual metal products (i.e. those that did not settle into the metal layer during the operation) and thus would be more leach resistant. In addition, there is currently no criteria in the TCLP procedure for iron or cobalt.

Phosphate additive - The purpose of the phosphate additive tests was to generate a low melting glass (e.g. 800°C) in order to reduce the amount of zinc that may be volatilized in the process. It would appear that in the care of the epoxy primer, two glass phases were produced. One is a phosphate glass as evidenced by the increased P concentration from the silicate-1100°C to the phosphate-800°C product extracts (both tests had about '0% sodium carbonate added). The behavior of phosphorus was counter to that of silicon which decreased in concentration. Boron also increased indicating that it may be associated with the phosphate phase while Sr, Na, Mn, Mg, Fe, Ca, Ba and Al decreased indicating an association with the silicate phase. Thus, it would appear that a 50% phosphate frit additive was not sufficient to tie up all the barium and did not lead to a non-hazardous ash (barium concentration in TCLP extract of 300 ppm).

The phosphate additive test with the green ash did not generate a non-hazardous product since the extract chromium level was greater than 5 ppm (Table 6-4). However, when compared to the silicate-1100°C

test, the extract level was somewhat lower (9.2 versus 13.8 ppm) indicating that some chromium went into the phosphate glass. However, other elements (B,Ba,K,Mg,Si,Sr) increased in leachability again suggesting that another glass phase (silicate) may have been produced. A greater amount of phosphate additive would have been necessary to tie up all the chromium.

6.4 Quality assurance/Quality Control Results

This section summarizes the results obtained for the quality control samples inserted by Arthur D. Little, Inc. throughout the program in the samples sent to Barringer Magenta, Ltd. In addition, results of the blank TCLP extracts, spiked TCLP extracts and interlab comparison of extracts analyzed for barium and chromium are presented. All of the quality control data is given in Appendix C.

6.4.1 Replicate Measurements

Solids - Replicate analysis of ash solids generally showed good repeatability (0 to 16% relative standard deviation [RDS]), except for Cr, Hg, Mo and Zr (see Table 6-7). The data (Table C-1, Appendix C) for chromium showed that this poor repeatability was at the hundred ppm level (less than 50 versus 147 ppm for duplicates), for molybdenum at 40 ppm level (less than 20 versus 40 ppm for duplicates), for mercury at the 36 ppb level (18 versus 36 ppb for duplicates) and for zirconium at the 13 ppm level (6 versus 13 ppm for duplicates). These data are understandable in view of the proximity of the levels to the detection limits and indicate that substantial variability can occur for the chromium data at the hundred ppm level for the solid analyses. Thus for samples which contain on the order of a hundred ppm of Cr, these cannot be distinguished from below detection. The other elements (Mo, Hg, Zr) were not observed in any of the ash samples to a great extent.

Solutions - Replicate analyses of solutions (Table 6-7 and Tables C-2 and C-3) showed repeatability of between 0 and 31% for all elements analyzed. This wide range was expected due to variations in concentration levels in solution which brought some elements near their detection limits.

6.4.2 Standards

Solids - Recovery of blind solid standards (Table 6-8 and Table C-4) was generally between the range of 74 to 110% except for the elevated recovery values observed for chromium (153%), sodium (182%) and selenium (163%). The elevated chromium recovery may be partly due to the repeatability problem associated with analysis of concentrations of hundreds of ppm (i.e. 300 ppm). The high recovery of sodium was a problem in these analyses and suggests using caution for any of the solids data at several thousand ppm levels. The high selenium recovery was not observed by Barringer Magenta Ltd. in their internal standards (NBS 1646) which showed 100% recovery at 0.06 ppm). This high recovery is not a problem in interpretation of data since little Se was found in any solids.

TABLE 6-7
SUMMARY OF QUALITY CONTROL DATA- DUPLICATES

Ana	lyte	Ash Solids (%RSD)	TCLP Extract (%RSD)	TCLP Extract (%RSD)	TCLP Extract (%RSD)
		^ -		10.5	
A1		-0.5	4.9	10.5	-20.7
As	*	6.1			
В			-8.7	3.1	-4.0
Ba	*	1.6	-27.6	30.7	-5.2
Вe		-2.9		-20.7	-19.4
Ca		16.3	-1.4	2.6	-4.6
Cđ	*				
Co		-4.4			0.0
Cr	*	200.0			-1.9
Cu		0.0	-2.1		-1.5
Fe		2.7			
Hg	*	-66.7			
K		2.4	4.7	0.0	-28.6
Mg		5.3	0.0	3.1	0.0
Mn		-0.7	0.0	4.1	
Mo		200.0	0.0	0.0	0.0
Na		11.1	0.0	0.0	0.0
Ni	**	-4.5		0.0	• • • • • • • • • • • • • • • • • • • •
P		0.6		2.7	-6.7
Pb	*	-3.5		,	3.7
Se	*	0.0			
Si		5.3	-0.8	8.2	-5.9
Sr		4.0	-2.9	7.4	0.8
Th				0.0	-200.0
Ti		5.7		0.0	200.0
V		0.0		0.0	2.0
Zn		6.6	-31.6	4.3	5.1
Zr		-73.7	- 52.0	٠+٠٠	٦.١
TOC		13.2			
		10.4			

TOC - Total Organic Carbon *RSD - Percent Relative Standard Deviation * - TCLP element ** - proposed TCLP element

TABLE 6-8 SUMMARY OF QUALITY CONTROL DATA - STANDARDS

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Percent Recovery NBS NBS NBS NBS Water Water Analyte Ash Water A1 82.1 110.3 As 119.6 88.7 115.2 93.5 Ba 107.5 91.1 97.9 Вe Ca 91.0 100.0 Cd Co 73.9 Cr 153.1 529.4 176.5 117.6 91.5 105.6 77.8 Cu 90.9 Fa 98.0 79.5 136.4 Hg 75.0 K 94.7 91.6 Mg 81.1 96.8 96.8 96.8 Ma Mo 126.3 182.4 Na Ni 101.6 109.1 127.3 P Pb 76.0 Se 163.1 Si 102.2 Sr 87.2 92.9 94.6 Th 85.0 Ti 103.9 88.7 V 97.0 79.2 118.9 106.8

64.0

34.9

52.3

Zn

^{*} TCLP element

^{**} Proposed TCLP element

Solutions- Recovery of blind liquid samples (Table 6-8) generally ranged between 78 and 136% except for chromium (117 to 529 %) and zinc (35 to 64 %). The data for chromium was at 0.017 ppm which is 1.7 time the detection limit of 0.01 ppm. Thus this variability in the recovery can be associated with the poor repeatability at concentrations near the detection limit and at higher levels (31% at 0.1 ppm). The low recovery data for zinc was not a major issue in the data since Zn is not a TCLP metal. In addition, at higher concentrations (e.g. solids data) recovery was 107%.

6.4.3 Blanks

The concentration of all analyzed elements (except those listed in Table 6-9) in the blank TCLP extracts were below detection. For those shown in Table 6-9 where detectable levels were observed in the blanks, barium is the only one of regulatory importance. Thus levels of barium in extracts in the range of 0.022 to 0.167 ppm may be due to contamination (e.g., from the filters or the sodium hydroxide or acetic acid solutions used to prepare the extraction fluid). This level of contamination is much smaller than the TCLP criteria for barium (100 ppm) and thus does not present a problem in the interpretation of the data. It should be noted that many extracts had barium levels less than 0.5 ppm and it is questionable if that level is due to the sample of due to contamination. However, major conclusions in this program were based on differences between the 0.5 ppm level and much higher levels (e.g 20 ppm). Other elements which were consistently present were sodium (1350 to 1660 ppm), strontium (0.015 to 0.016 ppm) and boron (0.004 to 0.097 ppm). The sodium was present as expected due to its addition to the extracting fluid (fluid #1). The other detected elements (Al, Ca, Fe, Mg, Mo and Zn) were present at very low levels (from one to seven times detection) and were not consistently observed. These data can thus reflect the variability associated with the repeatability of analyses done at the detection limit.

6.4.4 TCLP Spikes

For all the elements analyzed (Tables 6-10, 6-11 and Tables C-7 to C-14 in appendix C) all of the recovery data was between 72.4 and 133.3%. This is well within the TCLP criteria of between 50 to 150%. Thus, there was not requirement to analyze any of the samples via standard additions and none were. The good recovery observed for all the elements provides further support the accuracy of the data.

6.4.5 Interlab Comparison of Ba and Cr Data

Due to the importance of the barium and chromium data for TCLP extracts, sample of extracts were analyzed both a Arthur D. Little, Inc. and at Barringer Magenta, Ltd. and the data compared. The results are presented in Table 6-12. Excellent agreement between the two laboratories was observed with a relative percent difference of minus 6.7 to a plus 12.2 for barium and a minus 12.5 to plus 1.4 for chromium. These data further support the validity of the chromium data at concentrations higher than 0.45 ppm.

TABLE 6-9
SUMMARY OF QUALITY CONTROL DATA - BLANKS

Analyte [a]	Detection Limit [b]	Range Observed In [c]	Blanks Number of Samples Above Detection Limit
Al	0.01	0.01-0.04	3
В	0.0004	0.004-0.097	6
Ва	0.005	6.022-0.167	6
Ca	0.01	0.01-0.07	1
Fe	0.01	U.03	3
K	0.5	1	1
Nig	0.01	0.01-0.03	2
Mo	0.01	0.03-0.04	2
Na	[4]	1350-1660	[b]
Sr	0.001	0.015-0.016	4
Zr.	0.01	0.02-0.07	4

Note a: Only analytes where concentrations above detection were observed are listed.

Note b: Detection limit quoted are those reported by Barringer Magenta Limited. These values are not instrumental detection limits but rather values based upon their working experience.

Note c: Data are in ppm. Range does not include blanks which were below detection.

Note d: Sodium was added to the TCLP extracts.

TABLE 6-10 SUMMARY OF QUALITY CONTROL DATA - TCLP SPIKES

	Recovery of Spikes (%) [a]					
Analyte	Green	(PUP) (b)	Ashes [c]	Primer [d]	Ashes	

Al	96.7	93.	9 97.7	94.8	100.4	
В	100.2	82.				
Ba	98.0	65.		•	102.7	
Be	101.6	96.		95.4	95.3	
Ca	98.1	84.			97.6	
Cd	98.1	94.		123.5	99.0	
Co	109.4	73.4		77.0	94.2	
Cr	100.5	61.8		139.8	98.3	
Cu	96.2	93.6	97.9	96.7	97.7	
Fe	96.0	96.1	90.1	106.8	79.6	
K	126.0	62.2	114.9	85.6	128.2	
Mg	133,3	94.1	100.8	94.0	103.2	
Mn	96.8	99.3	96.0	91.3	100.5	
Mo	96.1	91.0		122.0	109.0	
Ní	101.5	95.0		115.2	117.7	
P	100.7	99.1	. 101.7	103.2	103.2	
Pb	100.0					
Si	91.7	70.2		-	94.4	
Sr	96.3	95.3		-	97.2	
Th	92.4	89.1		83.8	94.6	
Ti	97.3	96.7		96.5	94.7	
V	97.4	92.0		93.3	96.3	
Zn	98.9	90.7		-	93.6	
Zr	95.5	99.5	99.5	99.5	97.0	

[[]a]: In general, data are averages of recovery at two spiking levels.

[[]b]: 700 degree C ash TCLP extract
[c]: 1100 degree C ash TCLP extract
[d]: 1450 degree C glass(and metal) TCLP extract
[e]: 1450 degree C ash product

TABLE 6-11
SUMMARY OF QUALITY CONTROL DATA - TCLP SPIKES

	Recovery	of Spikes	(%) [a]
	BR/BL		Spent
Analyte	Mix	(Epox)	Filter
.1	10/ 0	101 1	07.6
A1	104.0	101.1	97.5
В	101.1	104.3	96.1
Ba	106.8	94.7	95.5
Be	101.5	102.7	93.7
Ca	105.2	100.5	120.4
Cd	103.7	96.8	89.5
Co	97.3	79.4	72.4
Cr	102.6	99.5	80.1
Cu	103.1	104.7	100.3
Fe	103.9	100.7	94.1
K	109.4	102.1	87.6
Mg	108.4	97.4	107.7
Mn	104.8	97.4	93.0
Mo	96.6	105.8	93.9
Ni	91.9	130.5	80.7
P .	108.8	102.8	107.0
Pb	100.0	100.0	100.0
Si	110.0	88.8	80.0
Sr	101.1	99.4	111.7
Th	97.7	111.7	81.4
Ti	1.5.0	102.4	95.6
V	105.1	100.8	86.7
Zn	103.0	110.6	•
Zr	104.7	100.0	100.3

Note a: In general, data are averages of recovery at two spiking levels.

BR/BL mix - Extract from brown/black CARC ash at 1000 degrees C.

Green(Epox) - Extract from green epoxy/polyamide ash at 1100 deg C.

Spent Filter - Extract from spray booth filter ash at 1000 deg C.

TABLE 6-12
SUMMARY OF QUALITY CONTROL DATA - INTERLAB COMPARISON

TCLP Sample	Bariu	m (ppm) [a]		Chron	Chromium (ppm) [a]		
Number	ADL	Barringer	RD(%)	ADL	Barringer	RD(%)	
150	96.6	106	-8.9	BDL	0.04	-	
151	0.7	0.68	2.9	3.6	3.7	-2.7	
152	801	775	3.4	BDL	BDL	-	
153	4.5	4.27	5.4	0.15	0.15	0.0	
154	698	678	2.9	BDL	0.35	-	
155	312	300	4.0	BDL	0.15	-	
156	3.8	3.74	1.6	9	9.27	-2.9	
157	1.5	1.42	5.6	14	13.8	1.4	
158	0.6	0.64	-6.3	3.6	3.63	-0.8	
159	BDL	0.167	-	BDL	BDL	-	
160	BDL	0.115	-	BDL	BDL	-	
161	BDL	0.353	-	0.16	0.18	-11.1	
162	21.3	13.7	13.9	0.12	0.12	0.0	

[[]a]: ADL data were derived at 493.4 nm or 233.5 nm for barium and 205.5 nm for chromium.

BDL = below detection limit. Detection limits for barium were 0.45(ADL) and 0.005(Barringer) ppm.

Detection limits for chromium were 0.064(ADL) and 0.01(Barringer) ppm.

RD = Relative Difference

7. CONCLUSIONS AND RECOMMENDATIONS

7.1 Introduction

The design and recommendation of a specific treatment (and possibly recovery) process for paint wastes needs to consider the following:

- · Specific composition of all the wastes to be treated,
- Technical feasibility with regard to the treatment and/or recovery goals, and
- · Economic issues.

The results of this laboratory research and development program address only some of these items. Specifically, the technical feasibility was shown for selected treatment and/or recovery options for a waste generated from the currently utilized paints but by no means from all paint wastes that may possibly be generated during depot painting and depainting operations. Economic issues were not addressed at all in this program, and this should be noted.

7.2 Ash Properties

On the basis of the results of tests performed under this program, it can be concluded that waste materials generated from the currently utilized paint pigments will contain a variety of elements which are of concern either from a regulatory aspect (i.e the material may be classified as hazardous via the TCLP criteria) or from a recovery aspect (i.e metal values). From a regulatory aspect, high concentrations of barium (in the epoxy primer) and chromium (in the various pigments, especially the green shades) are of most concern. The availability (leachability) of these elements from ash products, generated after thermal treatment (e.g. incineration, pyrolysis) of the waste material to destroy the hazardous organic components, will depend to a great extent on the temperature, time and possibly presence of oxygen in the thermal treatment process. It was shown in this program that thermal treatment (combustion at temperatures in the range of 700 to 1100°C) of epoxy primer material lead to an ash which yielded a TCLP extract which containing Ba in increasing concentration for the increased temperatures finally exceeding the criteria of 100 ppm for hazardous classification at 1100°C. Similarly, thermal treatment (combustion) of the green CARC paints produced an ash which yields a TCLP extract which contains Cr in increasing concentrations for increased temperatures finally leading up to levels which are about 72% of the criteria (5 ppm) at 1100°C for hazardous classification.

The options for treatment of waste generated from depot painting and depainting operations (discussed below) considered the following other type wastes which may also require treatment:

- Wastes generated from strip; ing operations using, for example, sand, steel, walnut hulls or plastic media; and
- Stripped paints containing pigments which were used prior to the issuance of criteria regarding the lead and chromate content and thus may contain these in the ash residues.

The production of a non-hazardous ash residue from thermal treatment processes therefore cannot be guaranteed based upon these two factors:

- (1) The previously used paints and primers will likely yield ashes which will fail the TCLP test based at least upon their chromate content; and
- (2) Certain thermal and oxidizing conditions will lead to ash from the primer and possibly some pigments yielding TCLP extracts having concentrations which approach or exceed the criteria for barium and/or chromium.

These factors indicate that treatment of both the organic and inorganic phase of the waste paints will be necessary to insure production of a non-hazardous residue.

There are, however, particular approaches which are more likely to generate a non-hazardous ash residue from thermal treatment of the paint waste. The include the following:

- Blending of waste materials in a manner which reduces concentrations of the barium and chromium components.
- Segregation of the waste to be treated so that it does not contain lead and chromate containing paints,
- Segregation of the waste to be treated so that it uses not contain the epoxy primer materials, and
- Thermal treatment at the lowest possible temperatures (below 1000°C) required to destroy the organics while not increasing the leachability of the barium and chromium compounds from the ash products.

One or more of the above approaches may be required and may be sufficient for treating the waste without the need for subsequent treatment of the ash residue. However, the segregating of paint wastes at a depot prior to treatment may not be feasible. Consequently, a more general approach which would encompass all variability in waste feed composition would consist of thermal treatment of the waste paints followed by glassification with or without recovery of metal values form the ash. These latter approaches are discussed in greater detail in the following sections.

A. ...

7.3 Thermal Treatment (Oxidizing) and Glassification

The technical feasibility of generating a non-hazardous residue from the ash generated after thermal treatment via glassification was shown by the results of this program. In particular, the glass generated using the silicate additive tested in the 1450°C produced a product which yielded a TCLP extract with reduced barium and chromium concentrations relative to products generated in previous tests. It should be noted that the TCLP test was performed on all materials including the glass products after grinding them to pass a 35 mesh (500 micron) screen. Since the original ash materials generally passed this screen without much grinding while the glassified products needed to be ground, this testing procedure was more stringent for the glass product. This procedure was used in order that glassification on a micro-scale would be tested rather than on a macro-scale. The laboratory tests performed were not aimed at optimizing the type and quantity of additives but rather were aimed to illustrate technical feasibility. Thus, if this approach was chosen, an optimization process would need to be performed to generate the proper silicate composition at the desired temperature (expected to be in the 1100 to 1200°C range). This overall approach (thermal treatment and glassification done in an oxidizing glassifier) would appear to be the simplest way of generating a non-hazardous residue. Laboratory tests also suggest the need for a pollution control device to collect the volatilized zinc oxide and other potential volatile species (e.g., S and P) that may be present.

This approach, however, does not consider the potential metal value in the waste.

7.4 Thermal Treatment (Reducing) Combined with Glassification and Recovery

7.4.1 Background

From a metals value (recovery) point of view, the ashes generated in this program contain three elements that may be of interest. These are cobalt (in green shades), chromium (in the green and brown shades) and zinc (in the epoxy primer). The relative value (per pound) of these metals in recent years has ranged at approximately \$5 to \$10 for cobalt, \$0.5 to \$1 for chromium/iron used in stainless steel and \$0.25 to \$0.75 for zinc. Thus, depending on the relative amount of each metal in the waste, the cobalt content may be of greatest interest followed by that of chromium and zinc.

The technical feasibility of separating cobalt, chromium and zinc from the ashed pigment residue and simultaneously generating a glassified non-hazardous residue was demonstrated in this program. Addition of glassification additives (i.e sodium carbonate/silica) together with a reducing agent (carbon) at 1450°C generated both glass and a metal phase containing the appropriate metals. A variety of technical issues were identified as being important for operation of the process. These were:

A high ratio of chromium to iron in the ash (greater than about 0.4 as shown in Figure D-1, Appendix D) may lead to a

metal phase with a high melting point (i.e. greater than 1550°C). In order to assure the presence of a molten metal phase which can be separated from the glass/slag phase, this ratio should be maintained below this level,

- The temperature of the process should be raised from that used in the laboratory test (1450°C) to about 1550°C.
- A collection device (e.g., condenser) for collection of volatilized zinc (possibly contaminated with zinc oxide) and other volatile metals elements (e.g., P) would be required.
- A collection device (e.g., condenser or scrubber) for removal of volatilized sulfur may be required.
- Inclusion of additives to maximize the possibility of generating acceptable metal products which can be used in other processes (e.g., reduction of phosphorous and sulfur content in the metal phase) would be required.

This test program showed that the metal phase generated from the epoxy primer ash product (molten Fe) contained phosphorus and (possibly) sulfur. The presence of phosphorus at 7 to 8 % in the iron (Table 6-6) resulted in a melting point lowering (see Table D-2, Appendix D). Additionally, some sulfur was detected (0.35%) but it cannot be concluded that this was associated with only the metal phase as barium (an element not expected to be part of the metal phase) presumably present as the oxide in the glass was also found in the area scanned by the scanning electron microscope. These two elements are of interest since for use in stainless steel processes, a scrap containing chromium and iron should have as little of these two elements (P and S) as possible.

This laboratory test program also showed that the metal phase generated from the green ash material consisted of a relatively rich chromium metal phase containing some iron and cobalt (76% Cr, 16% Fe and 6% Co, see Table 6-3). According to the Cr/Fe phase diagram (see Figure D-3 in Appendix D) the melting point of this phase (containing only Cr and Fe) would be on the order of 1700°C. The presence of cobalt (see the Co/Fe phase diagram, Figure D-4, Appendix D) may decrease the melting point further, but not markedly. Thus, in order to maintain a metal composition which will remain molten in the process, a monitoring of the input waste composition with regard to chromium and iron will be required. A ratio which is lower than about 0.4 (Cr/Fe) does not appear to be a major problem if the various types of paint wastes were mixed. The primer material contains a high amount of iron (15%) and no chromium. The green CARC paint ashes contained 17 to 20% chromium and 3 to 5% iron. The sand pigment contained 4% chromium and 3% iron. The brown pigment contained 12 % chromium and 9% iron. The black pigment contains no chromium but 14% iron. The green epoxy/polyamide ash contained about 2% chromium and 2% iron. Thus, a mixture of these paints (considering the relative quantity of primer used) is likely to produce a metal phase where the ratio of chromium to iron is lower than that observed for the green CARC ashes which were at the highest

extreme. In addition, introduction of other iron containing waste (i.e steel shot) may also help to reduce this ratio depending on its chromium content. In order for the Cr/Fe metal to be of interest to stainless steel producers, a desirable content would be in the 10 to 40% depending on the grade of stainless steel.

There are other important issues associated with the metal composition and they include the cobalt, sulfur and phosphorous content. A desirable target for recovery would be separation of the cobalt for its own intrinsic value as well as the fact that for the Cr/Fe phase to be of interest it must have as little cobalt as possible. The presence of S and P in the Cr/Fe phase also make it much less desirable from the stainless steel producers perspective. These facts suggest the need for subsequent treatment of the generated metal phase from the recuctive glassification process which is expected to produce a mixture of Cr/Fe/Co (possibly contaminated with P and S) in the metal phase.

The cobalt content of the ashes generated in this program suggest that the green CARC pigments contain most of it (1 to 2% of the ash). The metal phase generated from the green pigments contained about 6% (see Table 6-6). It should be recalled that the relative value of cobalt to chromium (or zinc) is about 10 to 1 so that a 1% content of cobalt is similar in value to about 10% of chromium (or zinc). If a mixture of paint wastes is treated, the expected composition in the metal phase would be decreased from that of the green primer by factors expecting to bring it below one percent but obviously dependent on the ratio of green CARC pigment in the waste or other sources.

As previously mentioned the phosphorus and sulfur content of the Cr/Fe metal phase should be minimized for it to be of interest. The levels of phosphorus would need to be reduced to trace levels (e.g less than 0.01 percent) while those for sulfur to lower levels (e.g less than 0.1 percent).

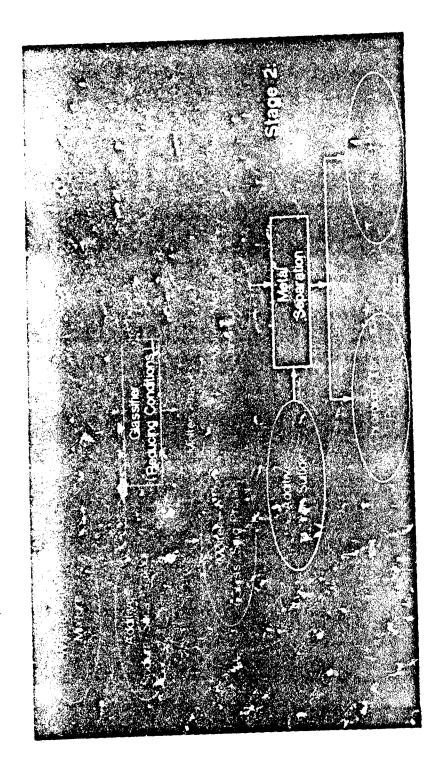
The zinc metal (possible contaminated with zinc oxide and other volatile metals present such as P) would be separated by volatilization from the reactor.

7.4.2 Treatment Approach

A possible approach which deals with the thermal treatment (reducing) combined with glassification and recovery aspects of the paint wastes is summarized in Figure 7-1. The first stage represents the thermal treatment (under reducing conditions) and generation of a glass and metal mixture, concurrent with the zinc recovery (and possible phosphorus volatilization). The second stage represents further treatment of the metal phase to produce products that may be of interest in other processes. The two stages are separated for clarity only and it is not implied that they would be independent although they could be.

In the first stage the wasts paint mixture is introduced into the reactor at the desired temperature (e.g., 1550°C). The waste contains a great deal of organic material (i.s., minimum of 50%) and this

FIGURE 7-1. GLASSIFICATION AND METALS RECOVERY APPROACH TO TREATMENT OF PAINT WASTES



Arthur D. Little, Inc.

provides the source of reducing agent for the system as long as the kinetics of volatilization and destruction in the gas phase are slower than decomposition in the molten phase so that carbon addition (as was done in the lab) may not be necessary. (Carbon has the advantage of not volatilizing from the melt). Additional additives are used which include the sodium carbonate and silica additives for glass making and calcium oxide which shifts the equilibrium for distribution of sulfur from the metal phase to the glass/slag phase (by making CaS) and minimizes the sulfur content of the product. The zinc present in the system is reduced to zinc metal and is volatilized and collected (e.g. in a condenser) from the gas phase. It is also envisioned that the reduced phosphrous would similarly volatilize and be selectively collected from the volatilized zinc. The reactor then contains the molten glass phase and a molten metal phase. At this point the metal phase can be separated, disposed, stored or directed to the second stage.

The second stage involves potentially refining the generated metal phase by first separation of the cobalt from it by addition of stoichiometric amount of iron sulfide to make cobalt sulfide which separates out as a separate phase from the melt. This final Cr/Fe phase may need to be cleaned for residual sulfur [and possibly phosphorus] (as was done previously using a glass/slag phase containing calcium oxide) depending on the residual content.

The second stage of this approach shown in Figure 7-1 is obviously not unique. There may be other processes that may be applicable (or already available) for treating a metal mix as is generated by the first stage. However, it does point out the issues of concern for the final products.

7.5 Other Relevant Issues

The above sections discussed the technical feasibility of generating a non-hazardous product via two different types of processes. Additional issues which require addressing include the following:

- Relative merit of the expected reduced pollution control costs involved with the reducing thermal treatment process versus the oxidizing thermal process.
- Relative cost of handling a larger mass (volume) of non-hazardous ash generated in the oxidizing thermal process (i.e. it includes the iron, chromium and cobalt oxides) versus the reducing thermal process.
- Relative cost of the increased amount of glassification additives required to generate a glass which contains the chromium, iron and cobalt oxides versus a glass without these materials in the reducing thermal treatment.

- The potential for a smaller (and more mobile) unit with the reducing thermal treatment system which can be moved from one depot to another.
- The reduced number of steps associated with the operation of the oxidizing thermal treatment versus the two stages for recovery.
- The benefits derived from recovering the chromium/iron cobalt and zinc materials.

7.6 Recommendations

The process for treating paint waste recommended for further investigation is thermal treatment. Initial experiments should be directed at testing an approach which would lead to an ash product which would be non-hazardous and thus not need to be further treated. If this approach is shown to be in appropriate, then further investigations are recommended of thermal treatment of the waste under reducing conditions with sodium carbonate, silica and other (to be determined) additives to generate both a glass and a metal phase. We would expect both these phases to be non-hazardous; with the metal phase either being disposed or upon further treatment generating saleable scrap materials.

Further investigation should be conducted on a pilot-scale level since many of the issues that need to be resolved cannot be done on a laboratory-scale unit. For the thermal treatment (without subsequent treatment of the ash) tests, these include the following:

- Destruction efficiency of the hazardous organic components present in the paint wastes,
- Generation of an ash residue which would pass the TCLP extract criteria (for both organic and inorganic components), and
- · Type of waste feed composition capable of being tested.

For the thermal treatment (reducing with glass generation and metal recovery) tests, these include the following:

- Destruction efficiency of hazardous organic components present in the paint wastes;
- Generation of glass and metal phases which will pass both the organic and inorganic analyses of the TCLP extracts;
- Effectiveness of the reducing glassifier for generating a reducing agent (i.e carbon and/or carbon monoxide) so that addition of carbon may not be needed;
- Effectiveness of the reducing glassifier for volatilizing zinc, phosphorus and sulfur components from the waste;

- Quality of the metal phase produced (specifically with regard to residual phosphorus and sulfur content);
- Feasibility of iron sulfide addition for separation of cobalt from the mixed chromium-iron-cobalt metal phase to generate both a chromium-iron and a cobalt sulfide saleable products; and
- Interactive effects of different waste compositions on the above issues.

APPENDIX A

CHEMICAL ANALYSIS DATA FOR SOLIDS AND EXTRACTS

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			_	_	

A-1 Percent Weight Loss For Various Paints Tested
A-2 Chemical Analysis Data For Ash Samples Generated
From the Epoxy Primer
A 3 Chemical Analysis Data for Ash Samples Generated

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- A-25 Solid and Extract Chemical Data for Gray/White Epoxy/Polyamide mix at Fast1000

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- A-27 Extract Chemical Analysis Data For Green Mix Ash Treated Under Various Conditions

TABLE A-1
WEIGHT LOSS FOR THE VARIOUS PAINT SAMPLES TESTED (a)

PAINT HEAT SAMPLE CONI (b)	TING DITION (c)	AMOUNT OF RESIDUE OVERALL(%)	AMOUNT REMAINING AFTER AIR DRYING (%)	AMOUNT REMAINING AFTER COMBUSTION (%)
EFOXY- FAST PRIMER RAME FAST	700 700 1000	45.3 46.3 39.5	87.6 87.6 87.6 37.6	52.8 52.9 45.1
383- FAST GREEN RAME CARC FAST RAME	700 700 1000 1000	37.9 36.4 36.5	72 72 72 72	52.7 50.6 50.7
GREEN RAME CARC FAST	700 1000	35.9 35.3	79.3 79.3 79.3 79.3	44.5
383- FAST SAND FAST CARC	700 1000	33.6 36.5	74.4 (d) 74.4	45.2 49
383- FAST BROWN RAMP CARC	1000	25.6 27.3	71.2 71.2	36 38.4
BLACK RAMP CARC	1000	33.3	79.4 79.4	42
SPRAY- RAMP FILTER RAMP	700 1000	57.0 55.4	DRY DRY	57 55.4
GREZN FAST EPOXY/ POLYAMIDE	1000	43.8	79.3	55.2
GRAY FAST EPOXY/ POLYAMIDE	1000	44.3	83.3	53.2
WHITE FAST EPOXY/ POLYAMIDE		25.9	78.6	32.9

Note a: Values for drying in air are averages of two data points.

Values for combustion are averages of from 4 to 8 data points. Note b: CARC - Chemical Agent Resistant Coating

Note c: Fast and Ramp refer to the heating rates at the respective temperatures in degrees centigrade.

Note d: One data point. Manufacturer specified 30.48 percent volatiles by weight.

Source: Arthur D. Little, Inc.

Arthur D. Little, Inc.

A-1

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TABLE A-2

CHEMICAL ANALYSIS DATA FOR ASH SAMPLES GENERATED FROM THE EPOXY PRIMER

	DETECTION	CONCENTRA	TION (MIC	ROGRAM PEI	R GRAM)
ANALYTE	LIMIT	RAMP	RAMP	FAST	FAST
	(ppm)	700	1000	700	1000
(SAMPLE	NUMBER)	9	10	11	12
Ag	0.5	BDL	BDL	BDL	3.3
Al		18000	22200	18900	22400
В	8	BDL	BDL	BDL	BDL
За		120000	137000	122000	134000
Be	0.05	2	2.03	2.1	2.3
Ca		8990	12200	9090	10800
Cd	1	BDL	BDL	BDL	BDL
Co	5	18	23	23	18
Cr		BDL	56	BDL	11
Cu	1	21	24	21	26
Fe		149000	177000	148000	188000
K	100	7700	10300	8100	10300
Mg		28100	33500	29200	33800
Mn		539	600	570	714
Mo	20	100	BDL	BDL	BDL
Na		1700	2000	1700	2100
Ni	5	90	90	91	92
P	50	33900	35000	34400	39400
Pb	5	425	560	435	155
Si		75200	88200	77200	88300
Sr	1	1120	1670	1230	1580
Th	1	BDL	BDL	BDL	BDL
Ti		261	279	254	265
V	1	10	12	8	12
Zn		108000	108000	102000	115000
Zr	5	64	37	13	48
As		8	•	8	-
Se		0.07	•	0.06	-
Hg (ppb)		36	•	34	-
TOC (%) [[a]	5.16	0.64	3.4	2.44

Note a: TOC - Total organic carbon

TABLE A-3

CHEMICAL ANALYSIS DATA FOR ASH SAMPLES GENERATED FROM THE 383-GREEN PAINT

ANALYTE	DETECTION LIMIT	CONCENTRA RAMP		ROGRAM PEI	
	(ppm)	700	1000	700	1000

(SAMPLE	NUMBER)	20	14	22	15
Ag	0.5	4.2	5.5	4.9	13.9
Al		3400			5600
В		263	208	260	230
Ba	27	150	157	[a]	129
Be	0.05	9.12	0.34	0.17	BDL
Ca		1160	1520	1210	1550
Cd	1	BDL	BDL	BDL	BDL
Co	5	14800	13100	14100	13200
Cr		205000	204000	202000	205000
Cu	1	234	225	227	245
Fe		27600	32000	27900	27900
K	100	600	700	600	700
Mg		70800	71000	70400	73600
Mn		46	50	47	55
Мо	20	90	BDL	100	50
Na		4500	5300	5000	5600
Ni	5	76	90	82	70
P	50	900	640	840	720
Pb	5	30	65	20	20
Si		186000	233000	185000	184000
Sr	1	9	9	10	10
Th	1	BDL	BDL	BDL	BDL
Ti		9150	11400	9140	9070
V	1	71	79	76	83
Zn		31800	29700	32500	32000
Zr	5		BDL		BDL
As		1.38	•	1.38	-
Se		0.07	-	0.05	•
Hg(ppb)		6	-	2	-
TOC(%)		0.2	0.16	0.24	0.16

Note a: Suspected error in analysis. Sample reported concentration below detection.

TABLE A-4

CHEMICAL ANALYSIS DATA FOR ASH SAMPLES GENERATED FROM THE FOREST GREEN PAINT

ANALYTE	DETECTION	CONCENTR	ATION (MI	CROGRAM PI	ER GRAM)
	LIMIT	RAMP	RAMP	FAST	FAST
	(ppm)	700	1000	700	1000
(SAMPLE	NUMBER)	21	. 16	23	17
Ag	0.5	3.4	4.2	3.4	12.2
AÎ		4410	4830	4380	4800
В		92.4	16.8	64.7	54.6
Ba		35	46	65	43
Be	0.05	BDL	BDL	BDL	BDL
Ca		1330	1520	1320	1630
Cd	1	BDL	BDL	BDL	BDL
Co	5	16500	12900	16800	15300
Cr		168000	172000	168000	185000
Cu	1	10	10	9	13
Fe		47900		48800	51100
K	100	800		700	900
Mg		73400		71300	76300
Mn		67		63	69
Mo	20	BDL		40	BDL
Na		6200		6200	7000
Ni	5	116	124	117	111
P	50	380	450	360	510
Pb	5	55	35	60	35
Si		204000	214000	214000	201000
Sr	1	9	8	9	9
Th	1	BDL	BDL	BDL	BDL
Tí		8620	8760	8670	8140
V	1	85	84	83	89
Zn		19700	16000	20200	20300
Zr	5	7	7	BDL	6
As		1	-	0.8	-
Se		0.07	-	0.05	-
Hg(ppb)		6	•	4	-
TOC (%)	0.02	0.44	0.36	0.24	BDL

TABLE A-5

CHEMICAL ANALYSIS DATA FOR ASH SAMPLES GENERATED FROM THE 383-SAND PAINT

ANALYTE	ANALYTE DETECTION		CONCENTRATION(PPM)		
	LIMIT	FAST	FAST		
	(ppm)	700	1000		
(SAMPLE	NUMBER)	32	33		
Ag	0.5	BDL	BDL		
Al		9580	10200		
В		83.3	59.8		
Ba		30	50		
Be	0.05	BDL	BDL		
Ca		1330	1610		
Cd	1	BDL	BDL		
Co	5	BDL	BDL		
Cr		36200	37800		
Cu	1	BDL	BDL		
Fe		33200	34200		
K	100	1400	1600		
Mg		2300	2610		
Mn		24	31		
Mo	20	190	15.7		
Na		7300	810 ₀		
Ni	5	36	38		
P	50	490	640		
Pb	5	BDL	BDL		
Si		279000	285000		
Sr	1	23	23		
Th	1	BDL	3		
Ti		124000	110000		
V	1	24	52		
Zn	1	94	BDL		
Zr	5	BDL	BDL		
As		1.38	2.5		
Se		BDL	BDL		
Hg (ppb))	6	7		
TOC (%)		0.64	0.16		

TABLE A-6
CHEMICAL ANALYSIS DATA FOR ASH SAMPLES GENERATED FROM THE 383 BROWN PAINT

ANALYTE	DETECTION	CONCENTRA	ATION (PPM)
	LIMIT	RAMP	FAST
	(PPM)	1000	1000
(SAMPLE	NUMBER)	26	28
Ag	0.5	2.7	9.9
Al		6160	6100
B		BDL	25.7
Ba	0.05	136	134
Be		BDL	BDL
Ca		1390	1470
Cd	1 5	BDL	BDL
Co		11	29
Cr		121000	130000
Cu	1	10	13
Fe		92900	93500
K		1200	1200
Mg	20	64300	71900
Mn		1.23	122
Mo		BDI	BDL
Na Ni P	5 50	7800 45 460	8200 50 530
Pb	5	465	295
Si		246000	245000
Sr		16	17
Th Ti V	i 1	BDL 297 74	BDL 272 77
Zn	5	376	70
Zr		BDL	BDL
As Se Hg (ppb)	0.02	-	0.9 BDL 4
TOC (%)		0.44	0.08

TABLE A-7

CMEMICAL ANALYSIS DATA FOR ASH SAMPLES GENERATED
FROM THE 383 BLACK PAINT

ANALYTE	DETECTION	CONCENTRATION (PP			
	LIMIT	RAMP	FAST		
	(myd)	1000	1000		
(SAMILE	NUMBER)	27	29		
Ag	0.5	BDL	2.2		
Αĺ		6510	6540		
В		BDL	16.8		
Ва		75	70		
Be	0.05	0.16	0.29		
Ca		1800	1980		
Cd	1	BDL	BDL		
Co	5	23	20		
Cr		95	137		
Cu	1	29	31		
F3		154000	137000		
K	100	1100	1100		
Mg		110000	97200		
Mn		295	301		
Mo	20	BDL	BDL		
Na		7300	7700		
Ni	5	45	51		
P	50	400	470		
Pb	5	BDL	BDL		
Si		272000	275000		
Sr	1	10	11		
Th	1	4	4		
Ti		321	303		
V	1	50	52		
Zn		451	239		
Zr	5	6	6		
As		•	3.1		
Se		-	0.02		
Hg (ppb)		-	7		
TOC (%)	0.02	BDL	0.08		

TABLE A-8

CHEMICAL ANALYSIS DATA FOR ASH SAMPLES GENERATED FROM THE SPENT SPRAY BOOTH FILTER SAMPLE

ANALYTE	DETECTION	CONCENTR	ATION (PPM)
	LIMIT	RAMP	RAMP
	(ppm)	700	1000
(SAMPLE	NUMLER)	52	53
Ag	0.5	BDL	2.9
Al		9690	13800
В		58.5	52.6
Ba		30600	30700
Be	0.05	0.72	1.02
Ca		5630	8250
Cd	1	BDL	BDL
Co	5	9570	31200
Cr		130000	124000
Cu	1	153	175
Fe		70300	64400
K	100	2300	3200
Mg		18200	17900
Mn		188	163
Мо	20	20	3 0
Na		11300	20300
Ni	5	75	108
P	50	8720	8090
Pb	5	375	400
Si		204006	193000
Sr	1	392	376
Th	1	BDL	BDL
Ti		5190	5200
V	1	63	73
Zn		46700	47600
Zr	5	22	18
As		2.7	2.2
Se	0.01	BDL	BDL
Hg (ppb)		147	68
TOC (%)		2.68	0.16

TABLE A-9

CHEMICAL ANALYSIS DATA FOR ASH SAMPLES GENERATED FROM THE GREEN, GRAY AND WHITE EXPOXY/POLYAMIDE PAINTS

ANALYTE		N CONCENTE	-	
-	LIMIT	GREEN	GRAY	WHITE
	(ppm)	FAST	FAST	FAST
		1000	1000	1000
/CAMDIE	NUMBER)	57	58	59
(SAMPLE	NONDER)	J/	ەر	29
Ag	0.5	BDL	BDL	BDL
Al		8690	8690	328C0
В		23	23.1	33.3
Ba		36.1	16.2	108
Ве	0.05	BDL	BDL	2.38
Ca		1980	1790	1390
Cd	1	BDL	BDL	BDL
Co	5	BDL	BDL	BDL
Cr		14500	96	93
Cu	1	73	9	26
Fe		14500	9720	889
K	100	500	500	270C
Mg		108	53	332
Mn		150	73	22
Мо	20	90	110	110
Na		700	400	700
Ni	5	38	34	56
P	50	300	250	280
Pb	10	BDI.	BDL	30
Si		287000	275000	85500
Sr	1	27	16	64
Th	1	44	37	40
Ti		217000	247000	439000
V	1	6	5	4
Zn		49	55	47
Zr	5	BDL	BDL	BDL
As		9	.4	8
Se	0.04	BDL	0.06	0.11
Hg (ppb)		42	7	7
TOC (%)	******		0.64	1.72

TABLE A-10

SOLID AND EXTRACT CHEMICAL ANALYIS DATA FOR EPOXY PRIMER AT RAMP 700 DEG C

AN	ALYTE	DET.LIMIT EXTRACT		TCLP EXTRACT	TCLP	EXCEEDS CRITERIA
SAI	MPLE #		9	37		
Ag Al B Ba		0.005	BDL 18000 BDL 120000	BDL 0.03 0.085 0.311	100	no
Be Ca Cd		0.0005	8990 BDL	BDL 126 BDL	1	no
Co		0.05	18	BDL	_	
Cr Cu Fe		0.01 0.008	BDL 21 149000	BDL BDL 0.02	5	no
K Mg Mn			7700 28100 539	9 105 0.92		
Mo Na Ni			100 1700 90	0.3 1410 0.2	15	[a] no
P Pb Si		0.05	33900 425 75200	14.8 BDL 53.3	5	no
Sr Th Ti V		0.05 0.005	1120 BDL 261	1.79 BDL BDL		
Zn Zr		0.005	10 108000 64	BDL 246 BDL		
As Se Hg	(ppb)		8 0.07 36	0.4 0.0035 1.8	[b] 5 [b] 1 [b] 200	no no no

Note b: Extract maximum level calculated using solid concentration

TABLE A-11

SOLID AND EXTRACT CHEMICAL ANALYIS DATA FOR EPOXY PRIMER AT FAST 700 DEG C

AN	ALYTE	DET.LIMIT EXTRACT		NTRATION TCLP EXTRACT	TCLP	EXCEEDS CRITERIA
SA	MPLE #		11	38		
Ag Al B Ba		0.005	BDL 18900 BDL 122000	BDL 0.04 0.104 0.38	100	no
Be		0.0005	2.1	BDL		
Ca Cd Co		0.01 0.05	9090 BDL 23	154 BDL BDL	1	no
Cr		0.01	BDL	BDL	5	no
Cu Fe		0.008	21	BDL		
re K			148000 8100	0.01 10		
Mg			29200	123		
Mn			570	1.19		
Mo Na			BDL	0.44		
Na			1700 91	1430 0.23	15	fal ma
P			3440C	13.5	ו כז	[a] no
РЪ		0.05	435	BDL	5	no
Si			77200	60.4		,
Sr Th		0.05	1230	1.26		
Tí		0.05 0.005	BDL 254	BDL BDL		
Ÿ		0.005	2.74	BDL		
Zn			102000	210		high
Zr		0.05	13	BDL		
As Se Hg	(ppb)		8 0.06 34	0.4 0.003 1.7	[b] 5 [b] 1 [b] 200	no no no

Note b: Extract maximum level calculated using solid concentration

TABLE A-12

SOLID AND EXTRACT CHEMICAL ANALYIS DATA FOR EPOXY PRIMER AT RAMP 1000 DEG C

ANALYTE	DET.LIMIT EXTRACT		TRATION TCLP EXTRACT	(PPM) TCLP CRITERIA	EXCEEDS CRITERIA
SAMPLE #		10	49		
			207		
Ag	0.005	BDL	BDL		
Al	0.01	22200	BDI		
В		BDL	0.077		no
Ва		137000	25.8		110
Be	0.0005	2.03	0.0006		
Ca		12200	58.2	_	no
Cd	0.01	BDL	0.05	_	110
Co	0.05	23	BDI		no
Cr	0.01	56	BCI	•	110
Cu	0.008	24	BDI		
Fe	0.01	177000	BDI		
K		10300			
Mg		33500	92.7 0.76		
Mn		600	0.70		
Мо		BDL	154		
Na		2000 90	BD1		[a] no
Ni		35000	12:	_	[4]
P	0.05	560			no
Pb	0.05	88200		_	
Sí		1670			
Sr	0.05	BPL			
Th	0.05	279			
Ti	0.005	12			
Ā	0.005	108000			
Zn	0.05	37	_		
Zr	0.05				

TABLE A-13

SOLID AND EXTRACT CHEMICAL ANALYIS DATA FOR EPOXY PRIMER AT FAST 1000 DEG C

		CONCE	NTRATION	(PPM)	
ANALYTE	DET.LIMIT	ASH	TCLP	TULP	EXCEEDS
	EXTRACT		EXTRACT	CRITERIA	CRITERIA
				•••••	•••••
SAMPLE #		12	42		
Ag	0.005	3.3	BDL	5	no
ΔĪ		22400	0.09		
В		BDL	0.096		
2 a		134000	43.3	100	no
Be	0.0005	2.3	0.0016		
Ca		10800	65.8		
Cq	0.01	BDL	BDL	1	no
Co	0.05	18	BDL		
Cr	0.01	11	BDL	5	no
Cu	0.008	26	BDL		
Fe	,	188000	BDL		
K		10309	30		
Mg		33800	75.5		
Mn		714	1.66		
Mo		BDL	0.44		
Na		2100	1420		
Ni		92	0.16	15 [a] no
P		39400	7.3		
Рb	0.05	155	BDL	5	no
Si		88300	85.2		
Sr		1580	4.19		
Th	0.05	BDL	BDL		
Ti	0.005	265	BDL		
V	0.005	12	BDL		
Zn		115000	320		
Zr	0.05	48	BDL		

TABLE A-14

SOLID AND EXTRACT CHEMICAL ANALYIS DATA FOR GREEN MIX AT FAST 700 DEG C

					ATION (PP			
ANA	LYTE	DET.LIMIT		FORS-GN	MIXTURE	TCLP EXTRACT		EXCEEDS CRITERIA
SAM	PLE #	•••••	22	23	25[c]			
Ag		0.005	4.9	3.4	4.15	3DL		
Al						0.84		
В			260	64.7	162.35	1.37		
Ba			BDL	65		0.138	100	no
Be		0.0005	0.17	BDL		0.0006		
Ca		•	1210	1320	1265	17.7		
Cd			BDL			BDL	1	no
Co		0.05	14100			0,41		
Cr		0.01	202000	168000	185000	0.11	5	no
Cu		0.008	227	9	118	BDL		
Fe			27900	48800	38350	BDL		
K			600		650	4		
Mg			70400		70850	90.6		
Mn			47	63	55	0.08		
Mo			100	40	70	0.08		
Na			5000	6200	5600	1460		
Ni			82	117	99.5	BDL	15 [a] no
P			840	360	600	1.4		
РЬ		0.05	20	60	40	BDL	5	no
Si			185000	214000	199500	20.7		
Sr			10	9	9.5	0.163		
Th		0.05	BDL	BDL		BDL		
Ti		0.005	9140		8905			
V		0.005	76	83	79.5	0.013		
Zn			32500	20200	26350	0.42		
Zr		0.05	BDL	BDL		BDL		
As			1.38	0.8	1.09	0.0545	[b] 5	no
Se				0.05		0.0025		no
Hg ((ppb)		2	4	3	0.15	[Ъ] 200	no

Note b: Extract maximum level calculated using solid concentration

Note c: Calculated from the composition of component paints.

TABLE A-15

SOLID AND EXTRACT CHEMICAL ANALYIS DATA FOR GREEN MIX AT RAMP 700 DEG C

	CONCENTRATION (PPM)							
ANALYTE	DET LIMIT	r ash	ASH	ASH	TCLP	TCLP	EXCEEDS	
	EXTRACT	383-GN	FORS-GN	MIXTURE	EXTRACT	CRITERIA	CRITERIA	
SAMPLE #		20	21	24[c]	40			
Ag	0.005	4.2	3.4	3.8	BDL	5	no	
Al		3400		3905	0.52			
В		263	92.4	177.7	1.7			
Ba		150	35	92.5	0.182	100	no	
Be	0.0005	0.12	BDL		0.0009			
Ca		1160	1330	1245	20.6			
Cd	0.01	BDL	BDL		BDL	1	no	
Co	0.05	14800	16500	15650	0.6			
Cr	0.01	205000	168000	186500	0.12	5	no	
Cu	0.008	234	10	122	BDL			
Fe		27600	47900	37750	0.02			
K		600	800	700	5			
Mg		70800	73400	72100	108			
Mn		46	67	56.5	0.07			
Mo		90	BDL		0.1			
Na		4500	6200	5350	1480			
Ni		76	116	96	BDL	15 [ai no	
P		900	380	640	2.4	•		
РЪ	0.05	30	55	42.5	BDL	5	no	
Si		186000	204000	195000	19.1			
Sr		9	9	9	0.182			
Th	0.05	BDL	BDL		BDL			
Ti	0.005	9150	8620	8885	BDL			
V	0.005	71	85	78	0.014		•	
Zn		31800	19700	25750	0.52			
Zr .	0.05	BDL	7		BDL			
As		1.38	1			[b] 5	no	
Se		0.07	0.07				no	
Hg (ppb)		6	6	6	0.3	[Ъ] 200	no	

Note b: Extract maximum level calculated using solid concentration

Note c: Calculated from the composition of component paints.

TABLE A-16 SOLID AND EXTRACT CHEMICAL ANALYIS DATA FOR GREEN MIX AT FAST 1000 DEG C

ANALYTE		ASH 393-GN	ASH FORS-GN	TION (PPM ASH MIXTURE	TCLP	TCLP CRITERIA	
SAMPLE #		15	17	19[b]	45		
Ag Al B	0.005	13.9 5600 230	12.2 4800 54.6	13.05 5200 142.3	0.12 0.967		no
Ba		129		86	0.332	100	110
Be Ca	0.0005	BDL 1550 BDL	BDL 1630 BDL		BDL 8.77 BDL	1	no
ca	0.01 0.05	13200			0.55		
Co Cr	0.01	205000				5	no
Cr	0,008	245	13				
Fe		27900					
K		700					
Mg		73600					
Mn		55					
Mo		50			0.15 1460		
Na		5600				٠	[a] n'
Ni		70					1-,
P		720					no no
Рb	0.05	20					
Si		184000 10					
Sr	0.05		-		BDL		
Th	0.05 0.005						
Ti	0.005					•	
V 7	0.003	32000				5	
Zn Zr	0.05				BDL		

Note b: Calculated from the composition of component paints.

TABLE A-17

SOLID AND EXTRACT CHEMICAL ANALYIS DATA FOR GREEN MIX AT RAMP 1000 DEG C

	CONCENTRATION (PPM)						
ANALYTE	DET.LIMI	T ASH	ASH	ASH	TCLP	TCLP	EXCEEDS
	EXTRACT	383-GN	FORS-GN	MIXTURE	EXTRACT	CRITERIA	CRITERIA
SAMPLE #		14	16	18[b]	50		
۸	0.005	5 5	4.2	4.85	BDL	5	no
Ag Al	0.005	4800		4815		,	110
В		208		112.4			
Ba		157				100	70
Be.	0.0005	0.34	BDL	101.5	BDL	. 100	no
Ca	0.0005	1520	1520	1520	9.58		
Cd	0.01	BDL	BDL	1320	BDL	1	no
Co	0.05	13100		13000	0.37	-	110
Cr	0.01	204000		188000	1.56	5	no
Cu	0.008	225	10	117.5	BDL	,	110
Fe	0.01	32000		41200	BDL		
K	1		900	800	BDL		
Mg	-	71000	81400	76200	26.5		
Mn		50	69	59.5	0.01		
Mo		BDL	BDL	37.3	0.07		
Na		5300	6600	5950	1540		
Ni	0.05	90	124	107	BDL	15	al no
P		640	450	545	2.7		
Рb	0.05	65	35	50	BDL	5	no
Si		233000	214000		8.94	_	
Sr		9	8	8.5	0.095		
Th	0.05	BDL	BDL		BDL		
Ti	0.005	11400	8760	10080	BDL		
A	0.005	79	84	81.5	0.516		
Zn		29700	16000	22850	1.18		
Zr	. 0.05	BDL	7		BDL		

Note b: Calculated from the composition of component paints.

BDL - Below Detection Limit

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TABLE A-18 SOLID AND EXTRACT CHEMICAL ANALYIS DATA FOR SAND AT FAST 700 DEG C

	DET.LIMIT		TRATION (PPM) TCLP	EXCEEDS
ANALYTE	EXTRACT	Ron	EXTRACT	CRITERIA	
SAMPLE #		32	41		
	0.005	BDL	BDL	5	no
Ag	0.005	9580	1.77	-	
Al		83.3	0.105		
В		30	0.204	100	no
Ва	0.0005	BDL	BDL	200	
Be	0.0005	1330	8.69		
Ca	0.01	BDL	BDL	1	no
Cd	0.01	BDL	BDL	-	
Co	0.05 0.01	3620	BDL	5	no
Cr	0.01	BDL	BDL	3	
Cu	0.008	33200	BDL		
Fe		1400	4		
K Mg		2300	6.9		
Mn		24			
Мо		190			
Na		7300	1420		
Ni		36	BDL	15	[a] no
P		490	BDL		
Pb	0.05	BDL	BDL	5	no
Si		279000	7.05		
Sr		23	0.209		
Th	0.05	BDL	BDL		
Ti	0.005	124000	BDL		
V	0.005	24			
Zn	•	94			
Zr	0.05	BDL	BDL		
As		1.38			
Se	0.02	BDL			
Hg(ppb)		6	0.3	[Ъ] 200	no

Note b: Extract maximum level calculated using solid concentration

TABLE A-19
SOLID AND EXTRACT CHEMICAL ANALYIS DATA FOR SAND AT FAST 1000 DEG C

		CONCE	NTRATION	(PPM)	
ANALYTE	DET.LIMIT	ASH	TCLP	TCLP	EXCEEDS
	EXTRACT		EXTRACT	CRITERIA	CRITERIA
SAMPLE #		33	47		
Ag	0.005	BDL	BDL	5	no
Al		10200	0.47		
В		59.8	0.198		
Ва		95	0.213	100	no
Вe	0.0005	BDL	BDL		
Ca		1610	6.87		
Cd	0.01	BDL	BDL	1	no
Co	0.05	BDL	BDL		
Cr	0.01	37800	0.19	5	no
Cu	0.008	BDL	BDL		
Fe		34200	BDL		
K		1600	2		
Mg		2610	6.88		
Mn		31	0.03		
Мо		190	0.05		
Na		8100	1490		
Ni		38	BDL	15 (a] no
P		640	1.9		
Pb	0.05	BDL	BDL	5	no
Si		569000	4.42		
Sr		23	0.123		
Th	0.05	3	BDL		
Ti	0.005	110000	BDL		
Λ	0.005	52	0.272		
Zn		BDL	0.17		
Zr	0.05	BDL	BDL		
As		2.5	0.125	[b] 5	no
Se	0.02	BDL	0.001	[b] 1	no
Hg (ppb)		7	0.35		no

Note b: Extract maximum level calculated using solid concentration

TABLE A-20

SOLID AND EXTRACT CHEMICAL ANALYIS DATA FOR BROWN/BLACK MIX AT FAST 1000 DEG C

			CONCENTRA	ATION (PP	M)		
ANALYTE	DET, LIMIT	T ASH	ASH	ASH	TCLP	TCLP	EXCEEDS
	EXTRACT	383-BRN	383-BLK	MIXTURE	EXTRACT	CRITERIA	CRITERIA
SAMPLE #		28	29	31[c]	46		
Ag	0.005	9.9	2.2	6.05	BDL	5	no
Al		6100	6540	6320	0.32		
В		25.7	16.8	21.25	0.129		
Ba		134	140	137	0.306	100	no
Be	0.0005	BDL	0.29		BDL		
Ca		1470	1980	1725	9.98		
Cd	0.01	BDL	BDL		BDL	1	no
Co	0.05	29	20	24.5	BDL		
Cr	0.01	130000	137	65068.5	BDL	5	no
Cu	0.008	13	31	22	BDL		
Fe		93500	137000	115250	0.39		
K		1200	1100	1150	1		
Mg		71900	97200	84550	9,22		
Mn		122	301	211.5	0.03		
Mo		BDL	BDL		0.07		
Na		8200	7700	7950	1460		
Ni		50	51	50.5	BDL	15 (al no
P		530	470	509	0.6	•	•
Pb	0.05	295	BDL	•	BDL	5	no
Si		245000	550000	397500	4.12		
Sr		17	11	14	0.077		
Th	0.05	BDL	4		BDL		
Ti	0.CO5	272	303		PDL		
V	0.005	77	52	64.5	0.09		
Zn		70	239	154.5	0.07		
Zr	0.05	BDL	. 6		BDL		
As		0.9	3.1	2			no
Se	0.02	BDL	0.02	ERR	0.001	[b] 1	no
Hg (ppb)		4	7	5.5	C.275	[ъ] 200	no

Note b: Extract maximum level calculated using solid concentration

Note c: Calculated from the composition of component paints.

TABLE A-21

SOLID AND EXTRACT CHEMICAL ANALYIS DATA FOR BROWN/BLACK MIX AT RAMP 1000 DEG C

			CONCENTRA	ATION (PP	1)		
ANALYTE	DET.LIMI	r ash	ASH	ASH	TCLP	TCLP	EXCEEDS
	EXTRACT	383-BRN	383-BLK	MIXTURE	EXTRACT	CRITERIA	CRITERIA
SAMPLE #		26	27	30[Ъ]	51		
A	0.005		222	1 25	22.	-	
Ag	0.005				BDL		no
Al		6160			0.06		
В		BDL			0.123		
Ва	0 0005	136		105.5		100	no
Ве	0.0005	BDL		0.08			
Ca	0.01	1390		1595			
Cd	0.01	BDL			BDL	1	no
Co	0.05	11		_		_	
Cr	0.01	121000		60547.5		5	no
Cu	0.008	10					
Fe		92900					
K		1200					
Mg		64300					
Mn		123		209	0.04		
Мо		BDL	_		0.1		
Na		7800	7300	7550	1540		
Ni		45	45	45	BDL	15 [[a] no
P		460		430	0.°		
Pb	0.05	465	BDL	233	0.2	5	no
Si		246000	272000	259000	43.3		
Sr		16	10	13	0.097		
Th	0.05	BDL	4	2	BDL		
Ti	0.005	297	321	309	BDL		
V	0.005	74	50	62	0.217		
Zn		376	451	413.5	0.35		
Zr	0.05	BDL	6	3	BDL		

Note b: Calculated from the composition of component paints.

TABLE A-22

SOLID AND EXTRACT CHEMICAL ANALYIS DATA FOR SPENT SPRAY BOOTH FILTER AT RAMP 700 DEG C

ANALYTE	DET.LIMIT	CONCE!	NTRATION TCLP	(PPM) TCLP	EXCEEDS
	EXTRACT		EXTRACT	CRITERIA	CRITERIA
SAMPLE #		52	55		
Ag	0.005	BDL	BDL	5	no
Al		9690	0.48		
В		58.5	0.304		
Ва		30600	0.994	100	no
Be	0.0005	0.72	BDL		
Ca		5630	86.3		
Cd	0.01	BDL	0.08	1	no
Co	0.05	9570	0.45		
Cr	0.01	130000	BDL	5	no
Cu	0.008	153	BDL		
Fe		70300	0.06		
K		3300	6.7		
Mg		18200	54.6		
Mn		188	0.54		
Mo		20	0.09		
Na		11300	1760		_
Ni		75	0.11	15 [a] no
P		8720	48.4	_	
Pb	0.05	375	BDL	5	no
SI		204000	26.4		
Sr		392	0.741		
Th	0.05	BDL	BDL		
Ti	0.005	5190	BDL		
V	0.005	63	0.063		
Zn		46700	238		
Zr	0.05	22	BDL		
As		2.7	0.135	[b] 5	no
Se		BDL	0	[b] 1	no
Hg (ppb)		147	7.35	` '	no

Note b: Extract maximum level calculated using solid concentration

TABLE A-23

SOLID AND EXTRACT CHEMICAL ANALYIS DATA FOR SPENT SPRAY BOOTH FILTER AT RAMP 1000 DEG C

		CONCE	NTRATION	(PPM)	
ANALYTE	DET.LIMIT	ASH	TCLP	TCLP	EXCEEDS
	EXTRACT		EXTRACT	CRITERIA	CRITERIA
SAMPLE #		53	56		
Ag	0.005	2.9	BDL	5	no
Al		13800	0.1		
В		52.6	0.122		
Ba		30700	0.259	100	no
Be	0.0005	1.02	BDL		
Ca		8250	86.3		
ca	0.01	BDL	0.09	1	no
Co	0.05	31200	1.54		
Cr	0.01	124000	0.7	5	no
Cu	0.008	175	0.019		
Fe		64400	0.04		
K		3200	4.1		
Mg		17900	88.2		
Mn		163	0.29		
Mo		30	0.19		
Na		20300	1940		
Ni		108	0.08	15 [a] no
P		8090	146		
Pb	0.05	400	BDL	5	no
Si		193000	6.29		
Sr		376	1.69		
1h	0.05	EDL	0.07		
Tí	0.005	5200	BDL		
V	0.005	73	189		
Zn		47600	38.7		
Zr	0.05	18	BDL		
As		2.2	0.11	[Ъ] 5	no
Se	0.04	BDL	BDL	[b] 1	no
Hg (ppb)		68		[ъ] 200	no

Note b: Extract maximum level calculated using solid concentration

TABLE A-24

SOLID AND EXTRACT CHEMICAL ANALYIS DATA FOR GREEN EPOXY/POLYAMIDE AT FAST 1000

			CONCE	NTRATION	(PPM)	
AN	ALYTE	DET.LIMIT	ASH	TCLP	TCLP	EXCEEDS
		EXTRACT		EXTRACT	CRITERIA	CRITERIA
SA	MPLE #		57	61		
		0.005	PDI	BDL	5	
Ag Al		0.003	BDL 8690	0.79	ر	no
B			23	0.79		
Ba			36.1	0.227	100	20
Be		0.0005	BDL	BDL	. 100	no
Ca		0.0003	1980	2.17		
Cd		0.01	BDL	BDL	1	no
Co		0.05	BDL	BDL	1	110
Cr		0.01	14500	BDL	5	no
Cu		0.008	73	0.049	,	110
Fe		0.01	14500	BDL		
K		0.01	500	4.2		
Mg			108	0.39		
Mn			150	0.41		
Мо			90	0.02		
Na			700	1490		
N1		0.05	38	BDL	15	al no
P		0.5	300	BDL	,	
Pb		0.05	BDL	BDL	5	no
Si			287000	5.06		
Sr			27	0.176		
Th		0.05	44	BDL		
Ti		0.005	217000	BDL		
V		0.005	6	BDL		
Zn			49	0.11		
Zr		0.05	BDL	BDL		
As			9	0.45	[b] 5	no
Se		0.04	BDL	BDL	[b] 1	no
Hg	(ppb)		42	2.1		no

Note a: Proposed criteria levels

Note b: Extract maximum level calculated using solid concentration

TABLE A-25

SOLID AND EXTRACT CHEMICAL ANALYIS DATA FOR WHITE AND GRAY GREEN EPOXY/POLYAMIDE AT FAST 1000

			CONCENTR	ATION (PP	M)		
ANALYTE	DET.LIMIT	ASH	ASH		TCLP	TCLP	EXCEEDS
	EXTRACT	GRAY	WHITE	MIXTURE	EXTRACT	CRITERIA	CRITERIA
	•••••	• • • • • •					
SAMPLE #		58	59	[c]	62		
A -	0.005	BDT	POT		BDL	5	
Ag	0.005	BDL 8690	BDL 32800			3	no
Al B		23.1	33.3	28.2			
Ba.		16.2	108	62.1		100	
Be	0.0005	BDL	2.38	1.18	BDL	100	no
Ca	0.0003	1790	1390	1590	6.68		
Cd	0.01	BDL	BDL	1370	BDL	1	no
Co	0.05	BDL	BDL		0.15	*	110
Cr	0.01	96	93	94.5		5	no
Cu	0.008	9	26	17.5		•	
Fe		9720	889				
K		500	2700	1600	7.4		
Mg		53	332	192.5	0.76		
Mn		73	22	47.5	0.07		
Мо	0.02	110	110	110	BDL		
Na		400	700	550	1480		
Ni		34	56	45	0.08	15	[a] no
P	0.5	250	280	265	BDL	·	
Pb	0.05	BDL	30	15	BDL	5	no
Si		275000	85500	180250	5.15		
Sr		16	64	40	0.157		
Th	0.05	37	40	38.5	BDL		
Ti	0.005	247000	439000	343000	BDL		
V	0.005	5	4	4.5	BDL		
Zn		55	47	51	0.22		
Zr	0.05	BDL	BDL		BDL		
As		4	8	6	0.3	[b] 5	no
Se		0.06	0.11				no
Hg (ppb)		7	7	7	0.35		no
						- •	

Note b: Extract maximum level calculated using solid concentration

TABLE A-26 EXTRACT CHEMICAL ANALYSIS DATA FOR EPOXY PRIMER ASH TREATED UNDER VARIOUS CONDITIONS

			CONCE	NTRATION	(PPM) [a]		
		TCLP	TCLP	TCLP	TCLP	TCLP	TCLP
ANALYTE	DET.LIMIT			EXTRACT	EXTRACT	EXTRACT	EXTRACT
	EXTRACT	1000	1100	1450	1100	800	1450
	(ppm)	fast		fixed			e recovery
SAMPLE #		42	150	152	154	155	162
Ag	0.005	BDL	BDL	BDL		BDL	BDL
Al		0.09	0.03	0.36		0.08	1.62
В		0.096	0.175	0.222		17.5	0.038
Ba		43.3	106	775	678	300	18.7
Be	0.0005	0.0016	BDL	0.0008	BDL	BDL	BDL
Ca		65.8	36.1	84.6	83.2	32.1	1.33
Cd	0.01	BDL	0.01	BDL	0.06	0.02	0.02
Co	0.05	BDL	BDL	0.08	BDL	BDL	BDL
Cr	0.01	BDL	0.04	BDL	0.35	0.15	0.13
Cu	0.008	BDL	BDL	0.012	BDL	BDL	BDL
Fe		BDL	7.71	434	184	44.8	128
K		30	126	90.7	101	188	2.1
Mg		75.5	43.4	248	227	114	2.03
Mn		1.66	0.87	5.33	4.36	1.15	0.17
Mo		0.44	0.4	2.9	1.5	0.4	0.8
Na		1420	1400	17.8	421	4.4	1480
Ni		0.16	0.08	0.41	0.26	0.08	BDL
P		7.3	49.7	3.8	3.1	96.6	0.8
Pb	0.05	BDL					
Si		85.2	55.8	183	381	133	6.2
Sr		4.19	3.77	14.1	12.9	7.19	0.255
Th	0.05	BDL	BDL	BDL	BDL	BDL	BDL
Ti	0.005	BDL	BDL	BDL	BDL	BDL	BDL
V	0.005	BDL	BDI	0.036	0.028	BDL	BDL
Zn		320	94.2	630	279	122	3.92
Zr	0.05	BDL	BDL	BDL	BDL	BDL	BDL

NOTE a: Criteria for various metals in the TCLP extract are as follows: Ag (5 ppm), Ba (100 ppm), Cd (1 ppm)
Cr (5 ppm), Ni (15 ppm- proposed), Pb (5 ppm)

TABLE A-27

EXTRACT CHEMICAL ANALYSIS DATA FOR MIXED GREEN ASH
TREATED UNDER VARIOUS CONDITIONS

ANALYTE *	DET.LIMIT EXTRACT (ppm)	TCLP EXTRACT 1000 fast 45	TCLP	NTRATION TCLP EXTRACT 1450 fixed		TCLP EXTRACT 800 phosphate	TCLP EXTRACT 1450 recovery
Ag	0.005	BDL	BDL	BDL	BDL	BDL	BDL
Al	0.003	0.12	0.16	0.16		0.04	0.26
В		0.967	0.734	0.069		1.31	0.085
Ва		0.332	0.677	4.27		3.74	0.353
Be	0.0005	BDL	0.0017	BDL		0.0007	BDL.
Ca		8.77	6.89	1.11		0.31	0.25
Cd	0.01	BDL	BDL	BDL		BDL	0.02
Co	0.05	0.55	0.51	0.06	0.14	0.22	8.51
Cr	0.01	1.1ϵ	3.7	0.15	13.8	9.27	0.18
Cu	0.008	BDL	0.069	0.229	0.225	0.24	0.215
Fe		0.03	0.03	1.55	0.04	0.08	20.4
K		1	2.8	1.5	5.9	20.4	0.7
Mg		31.5	21.4	2.64	2.12	5.02	1.36
Mn	0.01	0.01	BDL	0.03		BDL	0.1
Mo	0.2	0.15	0.2	BDL	_	BDL	BDL
Na		1460	1400	1410		1430	1370
Ni	0.05	BDL	BDL	BDL	-	BDL	0.08
P		1.1	3.1	1	BDL	0.8	BDL
Pb	0.05	BDL					
Si		6.82	9.94	2.34		3.06	1.44
Sr	0.05	0.057	0.131	0.078		0.062	0.017
Th	0.05	BDL	0.06	BDL	BDL	BDL	BDL
Ti	0.005	BDL	BDL	BDL	0.006	BDL	BDL
V 7	0.005	0.544	0.453	BDL	0.064	0.16	BDL
Zn	0.05	0.55	0.38	3.77	2.1	1.88	0.68
Zr	0.05	BDL	BDL	BDL	BDL	BDL	BDL

NOTE a: Criteria for various metals in the TCLP extract are as follows: Ag (5 ppm), Ba (100 ppm), Cd (1 ppm)
Cr (5 ppm), Ni (15 ppm- proposed), Pb (5 ppm)

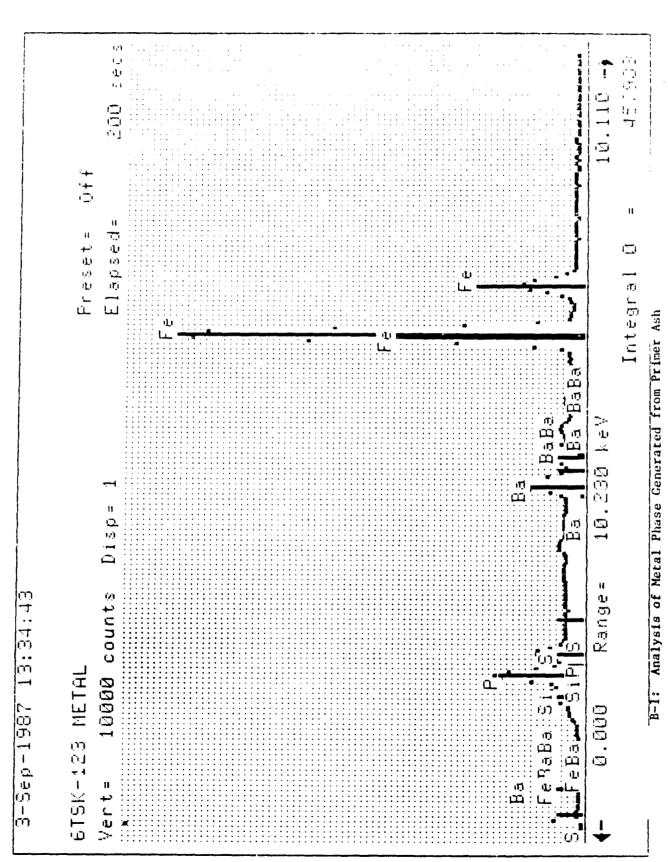
APPENDIX B

KEVEX/EDX ANALYSIS SPECTRA FOR SOLIDS AND SCANNING ELECTRON MICROGRAPHS

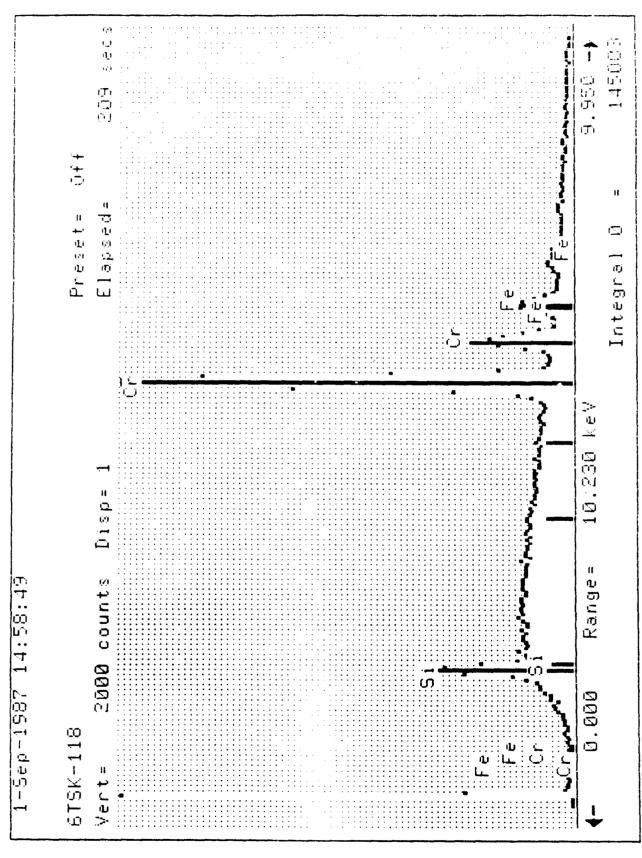
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	in the Metal Recovery Test
3-5	Scanning Electron Micrograph of Product Obtained
	in the Glassification and Recovery Test on Epoxy
	Primer Ash
3-6	Scanning Electron Micrograph of Product Obtained
	in the Glassification and Recovery Test on
	Creen CARC Ach



Arthur D. Little, Inc.



B-2: Analysis of Metal Phase Generated from Green Ash

B-3: Analysis of Glass Phase Generated from Green Ash in Metal Recovery Test

3-Sep-	p-1987	10:36:58			· · · · · · · · · · · · · · · · · · ·	
SAMPLE	E #123-	-GLASS				
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9-4: Analysis of Glass Phase Generated from Primer Ask in Netal Recovery Test



B-5: Scanning Electron Micrograph of Product Obtained in the Glassification and Recovery Test on Epoxy Primer Ash

B-5



Scanning Electron Micrograph of Product Obtained in the Glassification and Recovery Test on Green CARC Ash. (Flat area in center was gently sanded to expose glass material under metal coating.)

APPENDIX C
QUALITY CONTROL DATA

Arthur D. Little, Inc.

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TABLE C-1

QUALITY CONTROL DATA - ASH SOLID DUPLICATES

ANALYTE	SAMPLE A	SAMPLE B	% RSD
sample	11	35	
Ag	BDL	BDL	2.5
Al	18900	18800	-0.5
В	BDL	BDL	
Ba	122000	124000	1.6
Be	2.11	2.05	-2.9
Ca	9090	10700	16.3
Cđ	BDL	BDL	
Co	23	22	-4.4
Cr	BDL	147	
Cu	21	21	0.0
Fe	148000	152000	2.7
K	8100	8300	2.4
Mg	29200	30800	5.3
Mn	570	566	-0.7
Mo	BDL	40	
Na	1700	1900	11.1
Ni	91	87	-4.5
P	34400	34600	0.6
РЪ	435	420	-3.5
Si	77200	81400	5.3
Sr	1230	1280	4.0
Th	BDL	BDL	
Ti	254	269	5.7
V	8	8	0.0
Zn	102000	109000	6.6
Zr	13	6	-73.7
As	8	8.5	6.1
Se	0.07	0.07	0.0
Hg(ppb)	36	· 18	-66.7
TOC	3.4	3.88	13.2

TABLE C-2

QUALITY CONTROL DATA - TCLP EXTRACT DUPLICATES

ANALYTE	SAMPLE A	SAMPLE B	% RSD
sample	61	65	
Sampie	01	0.5	
Ag	BDL	BDL	
Al	0.79	0.83	4.9
P	0.06	0.055	-8.7
Ba	0.227	0.172	-27.6
Be	BDL	BDL.	
Ca	2.17	2.14	-1.4
Cd	BDL	BDL	
Co	BDL	BDL.	
Cr	BDL	BDL	
Cu	0.049	0.048	-2.1
Fe	BDL	BDL	
K	4.2	4.4	4.7
Mg	0.39	0.39	0.0
Mn	0.41	0.41	0.0
Mo	0.02	BDL	
Na	1490	1490	0.0
Ni	BDL	BDL	
P	EDL	BDL	
Рb	BDL	BDL	
Si	5.06	5.02	-0.8
Sr	0.176	0.171	-2.9
Th	BDL	BDL	
Ti	BDL	BDL	
V	BDL	BDL	
Zn	0.11	0.08	-31.6
Zr	BDL	BDL	

TABLE C-3

QUALITY CONTROL DATA - TCLP EXTRACTION DUPLICATION

ANALYTE	SAMPLE A		% RSD	SAMPLE A	SAMPLE B	% RSD	NOTE
sample	42	43		151	158	********	
Ag	BDL	0.007		BDL	0.006		
Al	0.09	0.1	10.5	0.16	0.13	-20.7	
В	0.096	0.099	3.1	0.734	0.705	-4.0	
Ba	43.3	59	30.7	0.677	0.643	-5.2	
Be	0.0016	0.0013		0.0017			
Ca	65.8	67.5	2.6	6.89	6.58	-4.6	
Cd	BDL	BDL		BDL	BDL		
Co	BDL	BDL		0.51	0.51	0.0	
Cr	BDL	BDL		3.7	3.63	-1.9	
Cu	BDL	BDL		0.069	0.068	-1.5	
Fe	BDL	BDL		0.03	BDL		
K	30	30	0.0	2.8	2.1	-28.6	
Mg	75.5	77.9	3.1	21.4	21.4	0.0	
Mn	1.66	1.73	4.1	BDL	BDL		
Но	0.44	0.47	6.6	0.2	BDL		
Na	1420	1420	0.0	1400	1400	0.0	
Ni	0.16	0.16	0 0	BDL	BDL		
P	7.3	7.5	2.7	3.1	2.9	-6.7	
Pb	BDL	BDL					
Si	85.2	92.5		9.94			
Sr	4.19	4.51	7.4	0.131		0.8	
Th	BDL	BDL		0.06	_		
Ti	BDL	BDL		BDL			
V 7	BDL	BDL		0.453			
Zn Zm	320	334	4.3			5.1	
Zr	BDL	BDL		BDL	BDL		

TABLE C-4

QUALITY CONTROL DATA - NBS TRACE ELEMENTS IN ASH STANDARD

ANALYTE	REF VALUE	SAMPLE %	REC
sample		34	
Ag	NA	BDL	
Al	140000	115000	82.1
В	NA	45	
Ba	1500	1330	88.7
Be	12	12.9	107.5
Ca	11100	10100	91.0
ca	1	BDL(1)	
Co	46	34	73.9
Cr	196	300	153.1
Cu	118	108	91.5
Fe	94000	92100	98.0
K	18800	17300	94.7
Mg	4550	4170	91.6
Mn	190	154	81.1
Мо	29	BDL(30)	
Na	1700	3100	182.4
Ni -	127	129	101.6
P	NA	2290	
Pb	72.4	55	76.0
Si	228000	233000	102.2
Sr	830	724	87.2
Th	24.7	21	85.0
Ti	8000	8310	103.9
V .	300	291	97.0
Zn	220	235	106.8
Zr	NA	193	
As	145	160	110.3
Se	10.3	16.8	163.1
Hg(ppb)	160	120	75.0

TABLE C-5

QUALITY CONTROL DATA - NBS TRACE ELEMENTS IN WATER STANDARD

ANALYTE	REF VALUE	SAMPLE	% REC	SAMPLE	* REC	SAMPLE	% REC
1-		44		64	******	163	
sample		44		04	•	163	
Ag	0.0028	BDL		BDL		BDL	
ΑĬ	NA	0.11		0.08		0.12	
В	NA	0.007		BDL		0.006	
Ba	0.046	0.053	115.2	0.043	93.5	0.055	119.6
Be	0.019	0.0173	91.1	0.018	94.7	0.0186	97.9
Ca	NA	25.1		24.7		23.9	
Cd	0.01	0.01	100.0	BDL		BDL	
Co	0.019	BDL		BDL		BDL	
Cr	0.017	0.09	529.4	0.03	176.5	0.02	117.6
Cu	0.018	0.019	105.6	0.013		0.014	
Fe	0.088	0.08	90.9	0.07	79.5	0.12	136.4
K	NA	BDL		BDL		1.3	
Mg	NA	7.78		7.18		7.48	
Mn					96.8	0.03	96.8
Мо	0.095		126.3	BDL		BDL.	
Na	NA	9		8.5		9.7	
Ni		0.06	109.1			0.07	127.3
P	NA	BDL		BDL		BDL	
РЪ	0.027	BDL		BDL			
Si	NA	BDL		BDL		BDL	
Sr	0.239	0.222	92.9		96.2	0.226	94.6
Th	NA	BDL		BDL		BDL	
Ti	NA	0.011		0.006		0.011	
V	0.053					0.063	
Zn			64.0		34.9	0.09	52.3
Zr	NA	BDL		BDL		BDL	

TABLE C-6

QUALITY CONTROL DATA - BLANK TCLP EXTRACTIONS [A]

ANALYTE sample	SAMPLE A	SAMPLE B	SAMPLE C	SAMPLE D	SAMPLE E	SAMPLE F
Ag Al			0.01		0.04	0.04
B Ba Be	0.007 0.022	0.008	0.022 0.076	0.004 0.086	0.097 0.167	0.019 0.115
Ca Cd Co Cr					0.03	
Cu Fe K			0.02		0.01 1 0.01	0.07
Mg Mn Mo Na	1420	1520	0.04 1660	0.03 1520	1350	[B]
Ni P Pb Si						
Sr Th Ti	0.015	0.016	0.016	0.015		
V Zn Zr	0.02	0.03	0.07	0.04		

NOTE A: ONLY VALUES ABOVE DETECTION LIMIT ARE SHOWN. ALL OTHER VALUES ARE BDL (BELOW DETECTION LIMIT)

EXTRACTION FLUID 2 HAS NO SODIUM ADDED TO IT.

SODIUM WAS NOT ADDED

EXTRACTION FLUID 1 HAS SODIUM ADDED TO IT.

TABLE C-7

QUALITY CONTROL DATA - SPIKED TCLP EXTRACT 39

ANALYTE	SAMPLE CONC	SPIKE LEVEL 1	SAMP+SPK 1	% REC	SPIKE LEVEL 2	SAMP+SPK 2	% REC
sample	39	SPK1	39SPK1		SPK2	39SPK2	******
Ag	0	0	0		0	0	
Al .	0.84	9.59	10.1	96.6	1.92	2.7	96.9
В	1.37	0.937	2.3	99.3	0.188	1.56	101.1
Ba	0.138	0.961	1.09	99.1	0.192	0.324	96.9
Be	0.0006	U.897	0.965	107.5	0.179	0.172	95.8
Ca	17.7	97.5	115	99.8	19.5	36.5	96.4
Cd	0	1.81	1.79	98.9	0.36	0.35	97.2
Co	0.41	1.72	2.2	104.1	0.34	0.8	114.7
Cr	0.11	1.87	1.95	98.4	0.37	0.49	102.7
Cu	0	1.89	1.83	96.8	0.378	0.361	95.5
Fe	0	22.6	21.9	96.9	4.52	4.3	95.1
K	4	16	23	118.8	3	8	133.3
Mg	90.6	15.3	111	133.3	3.06	93.9	133.3
Mn	0.08	2.02	2.07	98.5	0.4	0.46	95.0
Mo	0.08	5.03	4.87	95.2	1.01	1.06	97.0
Na	1460	40	1550		8	1490	37.0
Ni	0	1.94	1.9	97.9	0.39	0.41	105.1
P	1.4	37.1	39	101.3	7.4	8,8	100.0
Pb	0	1	1	100.0	0.1	0.1	100.0
S1	20.7	14.5	35.3	10C.7	2.9	23.1	82.8
Sr	0.163	2.02	2.1	95.9	0.403	0.553	95.8
Th	0	4.72	4.4	93.2	0.94	0.86	91.5
Ti	0	1.87	1.84	98.4	0.375	0.361	96.3
V	0.013	1.87	1.89	100.4	0.375	0.367	
Zn	0.42	1.89	2.27	97.9	0.38	0.367	94.4
Zr	0	1.98	1.9	96.0	0.4	0.38	100.0 95.0

NGTE: NO CALCULATION IS MADE WHEN SPIKE LEVEL IS TOO LOW FOR SAMPLE.
ALL BELOW DETECTION DATA (BDL) IS ASSIGNED A ZERO CONCENTRATION
FOR CALCULATION PURPOSES.

TABLE C-8

QUALITY CONTROL DATA - SPIYED TCLP EXTRACT 46

ANALYTE	SAMPLE CONC	SPIKE LEVEL 1	SAMP+SPK 1	% REC	SPIKE LEVEL 2	SAMP+SPK 2	% REC
sample	46	SPK1	46SPK1		SPK2	46SPK2	
Ag	0	0	0		0	0	
Al	0.32	9.66	10.2	102.3	1.93	2.36	105.7
В	0.129	0.93	1.08	102.3	0.186	0.315	100.0
Ba	0.306	0.915	1.24	102.1	0.183	0.51	111.5
Вə	0	0.905	0.918	101.4	0.181	0.184	101.7
Ca	9.98	97.4	112	104.7	19.5	30.6	105.7
Cd	0	1.75	1.78	101.7	0.35	0.37	105.7
Co	0	1.64	1.7	103.7	0.33	0.3	90.9
Cr	0	1.85	1.69	102.2	0.369	0.38	103.0
Cu	0	1.85	1.92	103.8	0.371	0.38	102.4
Fe	0.39	22	22.8	101.9	4.4	5.05	105.9
K	1	16	21	125.0	3.2	4	93.8
Mg	9.22	15	24.4	101.2	3.01	12.7	115.6
Mn	0.03	2.01	2.1	103.0	0.403	0.46	106.7
Мо	0.07	4.83	4.9	100.0	0.966	0.97	93.2
Na	1460	39	1500		7.9	1470	
Mi	0	1.79	1.8	100.6	0.36	0.3	83.3
P	0.6	37.3	40	105.6	7.5	9	112.0
Pb	0	1	1	100.0	0.1	0.1	100.0
Si	4.12	13.9	19.3	109.2	2.78	7.2	110.8
Sr	0.077	1.98	2.07	100.7	0.395	0.478	101.5
Th	0	4.72	4.7	99.6	0.94	0.9	95.7
Ti	0	1.86	1.96	105.4	0.373	0.39	104.6
Ā	0.09	1.87	2.07	105.9	0.374	0.48	104.3
Zn	0.07	1.75	1.83	100.6	0.351	0.44	105.4
Zr	0	1.92	2	104.2	0.38	0.4	105.3

NOTE: NO CALCULATION WAS MADE WHEN SPIKE LEVEL IS TOO LOW FOR SAMPLE.

ALL BELOW DETECTION DATA (BDL) IS ASSIGNED A ZERO CONCENTRATION
FOR CALCULATION PURPOSES.

TABLE C-9

QUALITY CONTROL DATA - SPIKED TCLP EXTRACT 56

ANALYTE	SAMPLE CONC	SPIKE LEVEL 1	SAMP+SPK 1	* REC	SPIKE LEVEL 2	SAMP+SPK 2	% REC
sample	56	SPK1	56SPK1		SPK2	56SPK2	
Ag	0	0	0		0	0	
Al	0.1	9.76	9.84	99.8	0.976	1.03	95.3
В	0.122	0.92	0.99	94.3	0.092	0.212	97.8
Ba	0.259	0.948	1.16	95.0	0.0948	0.35	96.0
Be	0	0.888	0.844	95.0	0.0888	0.082	92.3
Ca	86.3	98.7	187	102.0	9.87	100	138.8
Cd	0.09	1.77	1.76	94.4	0.177	0.24	84.7
Co	1.54	1.56	3.2	106.4	0.156	1.6	38.5
Cr	0.7	1.83	2.43	94.5	0.183	0.82	65.6
Cu	0.019	1.87	1.96	103.8	0.187	0.2	96.8
Fe	0.04	22.2	21.6	97.1	2.22	2.06	91.0
K	4.1	16.5	24	120.6	1.65	5	54.5
Mg	88.2	15.6	105	107.7		93	
Mn	0.29	2	2.31	101.0	0.2	0.46	85.0
Mo	0.19	4.84	4.98	99.0	0.484	0.62	88.8
Na	1940	40.2	1940		4.02	1960	
Ni	0.08	1.81	1.8	95.0	0.181	0.2	66.3
P	146	38.3	178	83.6	3.83	151	130.5
Pb	0	1	1	100.0	0.1	0.1	100.0
Si	6.29	14.5	20.4	97.3	1.45	7.2	62.8
Sr	1.69	1.97	3.89	111.7	0.197	2.09	
Th	0.07	4.81	4.6	94.2	0.481	0.4	68.6
Ti	0	1.84	1.81	98.4	0.183	0.17	92.9
V	C.189	1.84	1.968	96.7	0.184	0.33	76.6
Zn	38.7	1.79	39.6		0.179	38.4	
Zr	0	1.94	1.9	97.9	0.195	0.2	102.6

NOTE: NO CALCULATION WAS MADE WHEN SPIKE LEVEL IS TOO LOW FOR SAMPLE.
ALL BELOW DETECTION DATA (BDL) IS ASSIGNED A ZERO CONCENTRATION
FOR CALCULATION PURPOSES.

TABLE C-10

QUALITY CONTROL DATA - SPIKED TCLP EXTRACT 61

ANALYTE	SAMPLE CONC	SPIKE LEVEL 1	SAMP+SPK 1		SPIKE LEVEL 2	SAMP+SPK 2	% REC
sample	61	SPK1	61SPK1		SPK2	61SPK2	
Ag	0	0.019	0.02	105.3	0.002	0	
Al	0.79	10	10.6	98.1	1	1.83	104.0
В	0.06	0.907	0.966	99.9	0.091	0.159	108.8
Ba	0.227	1.01	1.2	96.3	0.1	0.32	93.0
Be	0	0.922	0.924	100.2	0.0922	0.097	105.2
Ca	2.17	98.9	99.6	98.5	9.89		102.4
Cd	0	1.87	1.85	98.9	0.19	0.18	94.7
Co	0	1.7		100.0	0.17		58.8
Cr	0	1.93	1.91	99.0	0.19	0 19	100.0
Cu	0.049	1.92	1.96	99.5	0.192	0.26	109.9
Fe	0	22.8	22.4	98.2	2.28	2.35	103.1
K	4.2	19	25	109.5			94.7
Mg	0.39	15.3	14.8	94.2			100.7
Mn	0.41	2.1	2.5	99.5			95.2
Mo	0.02	4.88	4.87	99.4	0.49	0.57	112.2
Na	1490	40.7	1530		4.1	1490	
Ni	0	1.94		103.1	0.19	0.3	157.9
P	0	37.9	38	100.3	3.8	4	105.3
Pb	0	1	1	100.0	5	5	100.0
Si	5.06	14.5	• •	98.9	1.45	6.2	78.6
Sr	0.176	2.01	2.15	98.2	0.201	0.378	100.5
Th	0	4.78	4.7	98.3	0.48	0.6	125.0
Ti	0	1.9	1.89	99.5	0.19	0.2	105.3
V	0	1.95	1.93	99.0	0.195	0.2	102.6
Zn	0.11	1.86	2.07	105.4			115.8
Zr	0	2	2	100.0	0.2	0.2	100.0

NOTE: NO CALCULATION WAS MADE WHEN SPIKE LEVEL IS TOO LOW FOR SAMPLE.
ALL BELOW DETECTION DATA (BDL) IS ASSIGNED A ZERO CONCENTRATION
FOR CALCULATION PURPOSES.

TABLE C-11

QUALITY CONTROL DATA - SPIKED TCLP EXTRACT 151

ANALYTE	SAMPLE CONC	SPIKE LEVEL 1	SAMP+SPK 1	% REC	SPIKE LEVEL 2	SAMP+SPK 2	% REC
sample	151	SPK1	151SPK1		SPK2	151SPK2	
Ag	0	0.011	0.02		0	0	
Al	0.16	10.2	9.72	93.7	1.02	1.12	94.1
В	0.734	1.05	1.74	95.8	0.107	0.808	69.2
Ba	0.677		1.55	89.6	0.103	0.72	41.7
Be	0.0317	0.975	0.974	99.7	0.0976	0.093	93.5
Ca	5.89	95.6	101	98.4	9.52	13.6	70.5
Cd	0	1.93	1.91	99.0	0.2	0.18	90.0
Co	0.51	2.15	2.7	101.9	0.2	0.6	45.0
Cr	3.7	2.03	5.6	93.6	0.2	3.76	30.C
Cu	0.069	1.99	2.01	97.5	0.202	0.25	89.6
Fe	0.03	23.4	22.8	97.3	2.31	2.22	94.8
K	2.8	20.2	26	114.9	2.1	3	9.5
Mg	21.4	14.9	35.8	96.6	1.53		91.5
Mn	0	2.18	2.15	98.6	0.21		100.0
Мо	0.2	5.1	5.4	102.0	0.5		80.0
Na	1400	39.3	1400		3.9	1400	
Ni	0	2.02	2	99.0	0.22		90.9
P	3.1	41.6	45	100.7	4	7	97.5
Pb							
Si	9.94			97.0	1.52		43.4
Sr	0.131	2.09	2.17	97.6	0.204	0.321	93.1
Th	0.06	4.26	4.2	97.2	0.42	0.4	81.0
Ti	0	1.99	1.96	98.5	0.2	0.19	95.0
v	0.453	2.02	2.45	98.9	0.208	0.63	85.1
Zn	0.38	2.04	2.38	98.0	0.24		83.3
Zr	0	2.02	2	99.0	0.2	0.2	100.0

NOTE: NO CALCULATION WAS MADE WHEN SPIKE LEVEL WAS TO LOW FOR SAMPLE.
ALL BELOW DETECTION DATA (BDL) IS ASSIGNED A ZERO CONCENTRATION
FOR CALCULATION PURPOSES.

TABLE C-12

QUALITY CONTROL DATA - SPIKED TCLP EXTRACT 152

ANALYTE	SAMPLE CONC	SPIKE LEVEL 1	SAMP+SPK 1	% REC	SPIKE LEVEL 2		% REC
sample	152	SPK1	152SPK1		SPK2	152SPK2	
					_		
Ag	0		0.02		0		22.2
Al	0.36				1.02	1.28	90.2
В	0.222			110.3		0.409	174.8
Ba	775				0.103		
Be	0.0008			97.5	0.0976		93.4
Ca	84.6		183			94.2	
Cd	0		1.97		0.2		
Co	0.08		2.1			0.2	
Cr	0	2.03				0.3	
Cu	0.012	1.99	2.01		0.202		93.1
Fe	434	23.4	459	106.8	2.31	440	
K	90.7	20.2	103	85.6	2.1	88	
Mg	248	14.9	262	94.0	1.53	252	
Mn	5.33	2.18	7.32	91.3	0.21	5.47	
Мо	2.9	5.1	8.2	103.9	0.5	3.6	140.0
Na	17.8	39.3	56	97.2	3.9	19	30.8
Ni	0.41	2.02	2.4	98.5	0.22	0.7	131.8
P	3.8	41.6	46	101.4	4	8	105.C
Pb							
Si	183	14.9	187		1.52	181	
Sr	14.1	2.09	15.5		0.204	13.9	
Th	0	4.26	4.1	96.2	0.42	0.3	71.4
Tí	0	1.99	1.95	98.0	0.2	0.19	95.0
V	0.038	2.02	2.04	99.1			87.5
Zn	630				0.24		
Zr	. 0			99.0	0.2		

NOTE: NO CALCULATIONS WERE INCLUDED WHERE SPIKE LEVELS WERE TOO LOW.
ALL BELOW DETECTION DATA (BDL) IS ASSIGNED A ZERO CONCENTRATION
FOR CALCULATION PURPOSES.

TABLE C-13

QUALITY CONTROL DATA - SPIKED TCLP EXTRACT 161

ANALYTE	SAMPLE CONC	LEVEL 1	SAMP+SPK 1		SPIKE LEVEL 2	SAMP+SPK 2	% REC
sample		SPK1	161SPK1		SPK2	161SPK2	
Ag	0	0.011	0		0	BDL	
ΑĬ	0.26	10.2	10.2	97.5	1.02	1.26	98.0
В	0.085	1.05	1.06	92.9	0.107	0.18	88.8
Ва	0.353	0.974	1.47	114.7	0.103	0.5	
Be	0	0.975	0.968	99.3			
Ca	0.25	95.6	95.5	99.6	9.52	9.29	95.0
Cd	0.02	1.93	1.92	98.4	0.2	0.2	90.0
Co	8.51	2.15	10.4	87.9	0.2	8.4	
Cr	0.18	2.03		99.0	0.2	0.37	95.0
Cu	0.215	1.99	2.19	99.2	0.202	0.41	96.5
Fe	20.4	23.4	43.3	97.9	2.31	22.3	32.3
K	0.7	20.2	25	120.3	2.1	3	1.09.5
Mg	1.36	14.9	16.5	101.6			00.0
Mn	0.1	2.18	2.21	96.8	0.21	0.3	95.2
Mo	0	5.1	5.3	103.9		0.6	120.0
Na	1370	39.3			3.9	1350	
Ni	0.08	2.02	2.1	100.0		0.3	100.0
P	0	41.6	43	103.4	4	4	100.0
Рb							
Si	1.44						
Sr	0.017	2.09		99.2	0.204		95.6
Th	0	4.26					
Ti	0	1.99					
V	0					0.24	
Zn	0.68	2.04			0.24		116.7
Zr	0	2.02	. 2	99.0	0.2	0.2	100.0

NOTE: NO CALCULATION WAS MADE WHEN SPIKE LEVEL WAS TOO LOW FOR SAMPLE ALL BELOW DETECTION DATA (BDL) IS ASSIGNED A ZERO CONCENTRATION FOR CALCULATION PURPOSES.

TABLE C-14

QUALITY CONTROL DATA - SPIKED TCLP EXTRACT 162

ANALYTE	SAMPLE CONC		SAMP+SPK 1		LEVEL 2	SAMP+SPK 2	% REC
sample	162	SPK1	162SPK1		SPK2		
Ag	0	0.011	BDL		0	BDL	
Al	1.62	10.2	11.3	94.9	1.02	2.7	105.9
В	0.038	1.05	1.02	93.5	0.107	0.154	108.4
Ва	18.7	0.974	19.7	102.7	0.103	19.2	
Be	0	0.975	0.94	96.4	0.0976	0.092	. 94.3
Ca	1.33	95.6	94.8	97.8	9.52	10.6	97.4
Cd	0.02	1.93	1.91		0.2		100.0
Co	0	2.15	1.9	88.4	0.2		100.0
Cr	0.13	2.03	2.09	96.6	0.2	0.33	100.0
Cu	0	1.99	1.92	96.5		0.2	99.0
Fe	128	23.4	145	72.6			86.6
K	2.1	20.2	26		2.1	5	138.1
Mg	2.03	14.9		100.5			105.9
Mn	0.17	2.18	2.27	96.3			104.8
Мо	0.8	5.1	5.8	98.0	0.5		120.0
Na	1480	39.3	1460		3.9		
Ni	0	2.02	2				
P	0.8	41.6	43	101.4	4	5	105.0
Pb							
Si	6.2	14.9	20.6	96.6			
Sr	0.255	2.09	2.24	95.0	0.204		
Th	0	4.26	4	93.9			95.2
Ti	0	1.99	1.88	94.5			95.0
V	0	2.02	1.95				96.2
Zn	3.92	2.04				4.14	
Zr	0	2.02	1.9	94.1	0.2	0.2	100.0

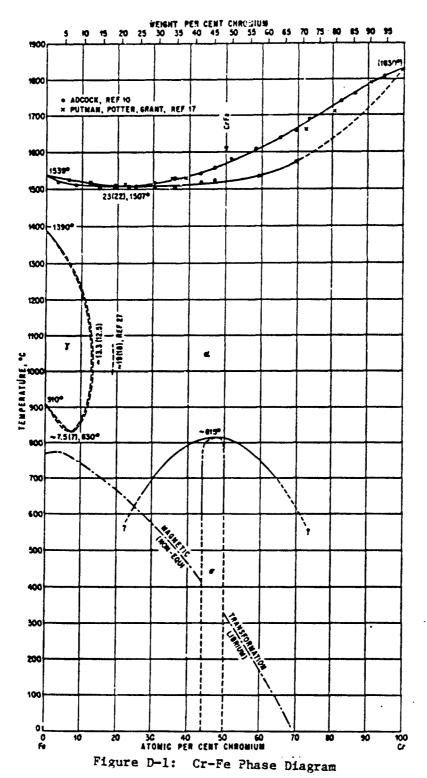
NOTE: NO CALCULATION WAS MADE WHEN SPIKE LEVEL WAS TOO LOW FOR SAMPLE.
ALL BELOW DETECTION DATA (BDL) IS ASSIGNED A ZERO CONCENTRATION
FOR CALCULATION PURPOSES.

APPENDIX D
PHASE DIAGRAMS

LIST OF FIGURES

FIGURE

D-1	Chromium/Iron Phase Diagram
D-2	Iron/Phosphorus Phase Diagram
D-3	Cobalt/Iron Phase Diagram
D-4	Cobalt/Cromium Phase Diagram



Source: M. Hansen, "Constitution of Binary Alloys." McGraw Hill Book Company, Inc. (1953).

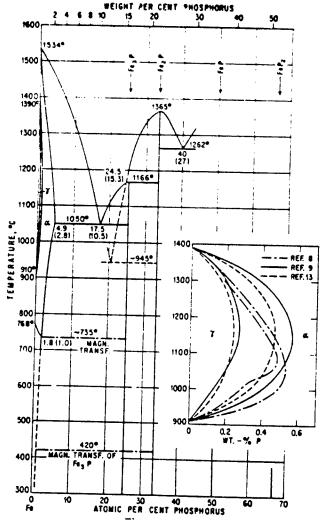


Figure D-2: Fe-P Phase Diagram

Source: M. Hansen, "Constitution of Binary Alloys." McGraw Hill Book Company, Inc. (1953).

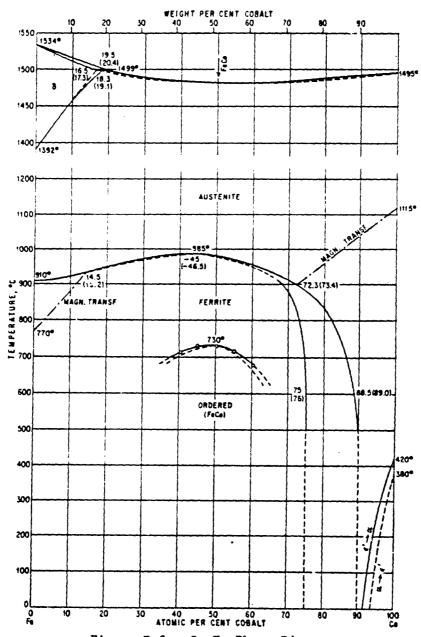
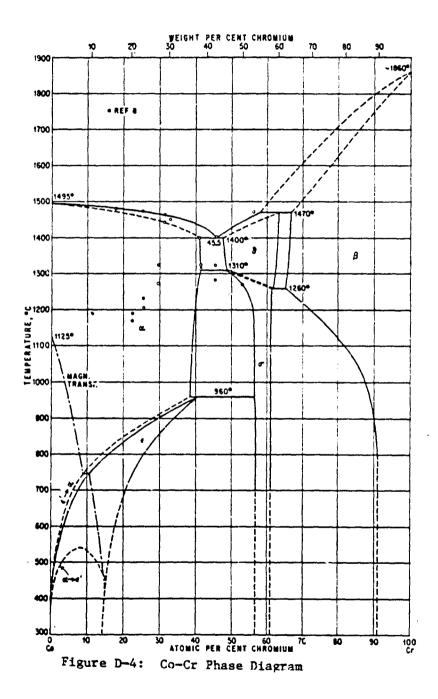


Figure D-3: Co-Fe Phase Diagram

Source: M. Hansen, "Constitution of Binary Alloys." McGraw Hill Book Company, Inc. (1958).



Source: M. Hansen, "Constitution of Binary Alloys." McGraw Hill Book Company Inc. (1958).

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